NASA-CR-54735

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report No. 7

For Quarter Ending April 15, 1965

EDITED BY E. E. HOFFMAN

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC

CINCINNATI, OHIO 45215

Requests for copies of this report
should be referred to:

National Aeronautics and Space Administration
Office of Scientific and Technical Information
Washington 25, D.C.
Attention: AFSS-A

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

QUARTERLY PROGRESS REPORT 7

(Covering the Period
January 15, 1965 to April 15, 1965)

Edited by
E. E. Hoffman
Project Manager

Approved by
J. W. Semmel, Jr.
Manager, Materials and Processes

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Under Contract NAS 3-2547

Technical Management
NASA - Lewis Research Center
T. A. Moss and R. L. Davies

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

CONTENTS

		Page
I	INTRODUCTION	1
II	SUMMARY	3
III	PROGRAM STATUS	5
	A. Component Evaluation Test Loop II	5
	B. Prototype Corrosion Loop Fabrication	15
	C. Alkali Metal Purification and Transfer Systems for the Prototype Corrosion Loop	39
	D. Prototype Corrosion Loop Components and Instrumentation	56
	E. Prototype Corrosion Loop Operation	80
	F. Refluxing Potassium Compatibility Tests	90
	G. Helium Analysis System	96
	H. Weld Contamination Study	96
IV	FUTURE WORK	99
	REFERENCES	101
	DISTRIBUTION LIST	103

ILLUSTRATIONS

Figure		Page
1	Manual and Hydraulic Cold Weld Pinch-Off Devices Used to Seal Cb-1Zr Alloy Tubing During Sectioning of Corrosion Test Loops.	8
2	Component Evaluation Test Loop II Showing Various Loop Temperatures During Test Operation and the Location of Chemical Analysis Samples	10
3	Microstructures of Cb-1Zr Tubing Following 2,650 Hours Exposure to Sodium Flowing at 7.5 fps	12
4	Cb-1Zr Bellows of Modified Hoke Valve Following Completion of Loop II Test. Isolation Valve Bellows (a) Unchanged During Test. Metering Valve Bellows (b) Shows Evidence of Some Distortion.	14
5	Cb-1Zr Bellows and Mo-TZM Alloy Plug of Loop II Metering Valve Following Disassembly of the Valve.	16
6	Magnet Flux Density vs Temperature of Component Evaluation Test Loop II Flowmeter Magnet Before and After 2,650-Hour Test.	17
7	Prototype Corrosion Loop Tube-in-Tube Helical Boiler.	19
8	Two Radiographs of the Boiler Inlet Region of the Prototype Corrosion Loop Showing the Plug Configuration	20
9	Mo-TZM Alloy Turbine Simulator Nozzle and Blade Assembly. . .	22
10	First Stage Turbine Simulator of Prototype Corrosion Loop . .	24
11	Turbine Simulator (Stages 2-10) of the Prototype Corrosion Loop Prior to Assembly. Nozzles and Blades: Mo-TZM. Tubing, Reducers, Casing and Miscellaneous Fittings: Cb-1Zr.	25
12	Potassium Duct (Top) and Sodium Duct (Bottom) of the Helical Induction Electromagnetic Pumps for the Prototype Corrosion Loop. All Parts Made of Cb-1Zr Alloy.	28
13	Potassium Surge Tank (6" OD) for the Prototype Corrosion Loop Partially Assembled.	29
14	Modified Hoke Valve and Associated Drive Components for the Prototype Corrosion Loop.	30
15	Stressed Diaphragm Pressure Transducer for the Prototype Corrosion Loop at Various Stages During Fabrication	31

ILLUSTRATIONS (Cont'd)

Figure		Page
16	Prototype Corrosion Loop Condenser and Fixture During Welding of Fin to Condenser Pipe.	33
17	Turbine Simulator, Condenser/Subcooler and Subcooler Reservoir of the Prototype Corrosion Loop Before (a) and Following (b) Application of the Iron Titanate Coating.	35
18	Preheater/Boiler/First Stage Turbine Simulator Assembly Wrapped with Cb-1Zr Alloy Foil Prior to Annealing.	36
19	Cb-1Zr Alloy Fin Specimen Coated with Iron Titanate. The Appearance of the Surface of the Coating at Low Magnification After the 1,000-Hour Evaluation Test is Shown in (a) and (b). Metallographic Cross Sections of the Coating Before Test (c) and After Test (d) are Also Included.	38
20	Simplified Schematic of Sodium and Potassium Purification and Transfer System for the Prototype Corrosion Loop.	40
21	Plan View Showing Spatial Arrangement and Interconnecting Lines Between Purification Systems, Transfer Systems and Loop	41
22	Sodium Hot Trap and Charge Tank	43
23	Sodium Purification System Showing Hot Trap and Charge Tank After Installation in Oven. Cover Panels Removed to Take Picture	44
24	Control Panel of Sodium Purification System for the Prototype Corrosion Loop.	45
25	Potassium Hot Trap for the Prototype Corrosion Loop Prior to Assembly. Type 316 Stainless Steel Container (10" Schedule 40 Pipe), Titanium Liner (0.040" Thick) and Zirconium Foil (0.012" Thick) are Shown.	48
26	Hot Trap for Purifying Potassium for the Prototype Corrosion Loop. Hot Trap has a Capacity of 50 Pounds of Potassium.	49
27	Potassium Distillation Unit for Purifying Potassium for the Prototype Corrosion Loop Test. Components of System Shown Prior to Assembly	50
28	Potassium Distillation Unit After Assembly.	51

Figure		Page
29	High Vacuum Potassium Still, Partially Assembled, Showing Annular Arrangement of Cb-1Zr Inner Tube and Type 310 Stainless Steel Outer Tube of Condenser. Potassium in the Annular Space Provides Heat Transfer for Condensing.	53
30	Assembled High Vacuum System for the Potassium Purification System.	54
31	Sodium and Potassium Transfer Systems for the Prototype Corrosion Loop After Installation in Oven Enclosure. Cover Panels Removed to Take Picture.	55
32	Alkali Metal Sampling Device.	57
33	Sensitivity of Prototype Loop Partial Pressure Analyzer	59
34	Vacuum Furnace for Calibrating W-3%Re/W-25%Re Thermocouple Wire .	63
35	Components of Thermocouple Calibration Vacuum Furnace Before Assembly.	64
36	Inside of Thermocouple Calibration Furnace Prior to Insertion of Mo-TZM Alloy Isothermal Block	66
37	Thermocouple Calibration Furnace Attached to the 24-Inch Diameter Getter-Ion Pumped Vacuum Chamber.	67
38	Thermocouple Bundle Used in Calibration of W-3%Re/W-25%Re Thermocouple Wire for the Prototype Corrosion Loop.	68
39	Difference Curve vs Temperature Obtained in Calibration of Two W-3%Re/W-25%Re Thermocouples Made from Wire Used to Instrument the Prototype Corrosion Loop.	71
40	Magnet Flux Density vs Temperature of the Flowmeter Magnets of the Prototype Corrosion Loop.	75
41	Deflection vs Load for the Prototype Corrosion Loop Cb-1Zr Alloy Valve Bellows	79
42	Schematic Diagram of the Prototype Corrosion Loop	82
43	Prototype Corrosion Loop Thermocouple Instrumentation Layout. . .	83
44	Cb-1Zr Alloy Refluxing Potassium Test Capsule Containing Mo-TZM Alloy Tubular Inserts in the Condenser Zone Following Completion of 2,500-Hour, 2000°F Test.	91

ILLUSTRATIONS (Cont'd)

Figure		Page
45	Potassium Reflux Capsule Components Following 2,500-Hour Test at 2000°F. Enlarged View of Cb-1Zr Capsule Wall at Liquid-Vapor Interface Shown on the Left.	94
46	Condensing Rates and Temperatures of Mo-TZM Refluxing Capsule Test #2 During the First 3,300 Hours of Operation.	95
47	Beckman Electrolytic Hygrometer and Oxygen Trace Analyzer to be Used for Monitoring Purity of Argon Cover Gas for Prototype Corrosion Loop	97

TABLES

Table		Page
I	Chemical Analyses Results Obtained on the Four Layers of Cb-1Zr Foil Used to Insulate the Top Heater Coil of Loop II During 2,650-Hour Test.	7
II	Results of Chemical Analysis on Specimens of Tubing from Component Evaluation Test Loop II Which Circulated Sodium at a Maximum Temperature of 2065°F for 2,650 Hours	11
III	Air Pressure Drop Test of Loop II Sodium Pump.	13
IV	Orifice Diameters for the Mo-TZM Alloy Nozzles for the Prototype Corrosion Loop Turbine Simulator as Determined by Air-Gauge Inspection	23
V	Results of Chemical Analyses of Cb-1Zr Alloy Welds After Heat Treatment	27
VI	Chemical Analytical Results on Sodium for the Prototype Corrosion Loop Following the Hot Trap Purification Treatment	47
VII	Prototype Loop Partial Pressure Analyzer Calibration I (3-3-65)	61
VIII	Comparison of Ion Gauge Partial Pressures and True Partial Pressures of Various Species in the Prototype Loop Chamber . .	62
IX	Characteristic Curve for W-3%Re/W-25%Re Based on Vacuum Calibration Data	73
X	Comparison of Reference Thermocouples and Optical Pyrometer Readings During Thermocouple Wire Calibration in Vacuum. . . .	74
XI	Prototype Loop Operation	88
XII	Weight Change Data Obtained on Specimens Following Cb-1Zr/Mo-TZM Refluxing Potassium Capsule Test.	93

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

I INTRODUCTION

This report covers the period, from January 15, 1965 to April 15, 1965, of a program to develop a Prototype Corrosion Test Loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The prototype test consists of a two-loop Cb-1Zr facility; sodium will be heated by direct resistance in the primary loop and will be used in a heat exchanger to boil potassium in the secondary, corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high-vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperature could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F
2. Superheat temperature, 2000°F
3. Condensing temperature, 1350°F
4. Subcooling temperature, 800°F
5. Mass flow rate, 20 to 40 lb/hr
6. Vapor velocity, 100 to 150 ft/sec
7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr ft^2

The development program includes the construction and operation of three Cb-1Zr test loops, each of which are being used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1,000 hours with liquid sodium at a maximum temperature of 2260° to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase sodium, forced-circulation loop to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers has completed 2,650 hours of scheduled testing. This loop was operated at a maximum temperature of 2065°F and a pump inlet temperature of 1985°F. The Prototype Corrosion Test Loop, a two-loop system, which has been designed and partially fabricated, will include a boiler, turbine simulator and condenser and in addition to the above components. This facility will be used to develop and endurance test (2,500 hours) the components required to achieve stable operation at the corrosion test design conditions.

The quarterly progress reports issued for this program will summarize the status of the work with respect to design considerations, construction procedures, and test results. Detailed topical reports will also be issued to describe each test loop. Additional topical reports will be prepared to cover such areas as materials specification, purification of potassium and sodium, and inert gas purification and analyses.

II SUMMARY

During the past quarter, the evaluation of Component Evaluation Test Loop II was substantially completed. The loop components performed in a satisfactory manner and both metallographic examination and chemical analyses of loop tubing indicated that no significant corrosion had occurred during the 2,650-hour test at 2000°F with liquid sodium.

All major subassemblies of the Prototype Corrosion Loop have been completed. Following annealing of the condenser - turbine simulator, this subassembly was shipped to Pratt & Whitney Aircraft where an iron titanate high emittance coating was applied to the Cb-1Zr alloy fins of the condenser. Final assembly of loop components should be completed by June 15.

Fabrication of the numerous alkali metal purification and transfer system components has been essentially completed. The sodium for the primary circuit of the Prototype Loop has been purified by hot trapping with zirconium foil and is ready for use. Purification of the potassium for the secondary circuit of the loop is underway.

Calibration of numerous components of the Prototype Loop, including the partial pressure analyzer, refractory alloy thermocouples, flowmeter magnets, and pressure transducer, has been completed. A detailed test plan for the Prototype Loop has been prepared and submitted to NASA for approval.

The 2,500-hour, 2000°F refluxing potassium test to determine the compatibility of Mo-TZM alloy in the condensing region of a Cb-1Zr alloy capsule has been completed and a preliminary evaluation performed. Weight change data indicate no significant compatibility problems. A second capsule test is being continued for a total of 5,000 hours to determine the effect of time on interactions in this three component system.

III PROGRAM STATUS

A. Component Evaluation Test Loop II

The evaluation of the Loop II test continued during this quarter and the various phases of the evaluation are summarized below:

1. Analysis of Sodium

It was indicated in the previous progress report (1) that the sample of sodium taken from Loop II following completion of the 2,650-hour test analyzed 5.3, 7.6, 4.9 and 5.8 ppm oxygen as determined by the mercury amalgamation method. Sodium samples taken during the draining of the loop were also submitted to General Atomic for determination of oxygen by the neutron activation method. Values of 394 $\pm$ 31 ppm and 409 $\pm$ 33 ppm were obtained by General Atomic. It was difficult to understand these results in light of the oxygen concentration determined by the amalgamation procedure and the sodium sample history, i.e., 2,650 hours in contact with Cb-1Zr alloy at a temperature of approximately 2000°F. After conducting a considerable number of analyses on the sodium samples returned to General Electric from General Atomic, none of which shed any light on the apparent discrepancy, General Atomic discovered a source of error in their procedure for analyzing for oxygen in sodium.

The analysis of sodium from oxygen by neutron activation using 14 Mev neutrons results in the reaction Na^{23} $(n,\alpha)F^{20}$. The half-life of F^{20} is 10.8 seconds and it decays with gamma emission in a manner similar to N^{16} which has a half-life of 7.6 seconds. The determination of oxygen depends on the O^{16} $(n,p)N^{16}$ reaction. The ability to distinguish between gamma radiation from F^{20} and N^{16} depends on the temporal resolving capability of the electronics in the detection system. It was discovered recently at General Atomic that the resolution of their detection system was not adequate to eliminate the F^{20} radiation and, consequently, the results for oxygen concentrations determined in the General Electric sodium samples were high. Subsequent studies at General Atomic have revealed that major difficulties have been encountered in the application of the fast neutron activation method for determining the oxygen concentration of sodium which do not exist in the application of this method to the determination of oxygen in potassium (2).

2. Disassembly of Loop Components

During the disassembly of the loop, four layers of the 0.002-inch thick Cb-1Zr alloy foil from the hottest region (2065°F) of the loop were removed and analyzed in order to obtain a sensitive indication of the quality of the Loop II test environment during the 2,650-hour test. With a half thickness of only 1 mil, even small amounts of oxygen pickup may readily be detected. The results of these analyses are given in Table I. The maximum oxygen increase (approximately 380 ppm) was detected in the outer foil as one would predict. A slight amount of nitrogen contamination of the outer foil is also apparent. These layers of foil as well as all others removed from loop components had the appearance and ductility of the pretest foil as determined by reverse bending to fracture.

The first step in removing actual loop components was to cut through the stainless steel can which formed the vacuum enclosure for the EM pump. In cutting up the loop proper, a procedure was used which permitted the maintenance of gas-tight seals of each section removed from the loop. This was accomplished by means of a cold-weld pinch-off device* which has a remote hydraulically actuated head which could be conveniently positioned in the many restricted areas where "cuts" were desired. Figure 1 shows the two models of the pinch-off device which have been used and a typical cold-weld seal in small diameter Cb-1Zr tubing. This technique not only gave helium leak tight seals which prevented reaction of the atmosphere with possible corrosion products but also precluded the possibility of introducing metallic particles into the loop, such as would have been the case if a hack saw had been used.

The sealed segments of the loop were then placed in the vacuum/purge welding chamber, where the pinch sealed ends were cut off under argon with a tubing cutter, and each section was examined to determine if any corrosion products or residual sodium were present. Those sections which showed evidence of residual sodium were transferred to the vacuum distillation unit for additional cleaning. The distillation unit used was described in an earlier report (3). Examination of all the loop components indicated that the initial distillation of sodium from the loop while still in place in the test chamber was quite successful with only a small amount of sodium remaining in the inlet and outlet tee fittings to the EM pump.

Following final cleaning in the distillation unit, all sections of the loop were carefully examined for evidence of mass transfer or corrosion. The only thing found was a fragile flake of dark material weighing less than 2 mg which gave a very weak spectrographic indication of silicon. This particle was found in the leg of the tee fitting which led to the bypass containing the metering valve. It is presumed that this particle was somehow introduced during the stripping procedure, however, the care taken to avoid such an occurrence would seem to preclude this.

----

* Kane Engineering Laboratories, Palo Alto, California.

TABLE I. CHEMICAL ANALYSES RESULTS OBTAINED ON THE FOUR LAYERS OF Cb-1Zr FOIL[1] USED TO INSULATE THE TOP HEATER COIL OF LOOP II DURING 2,650-HOUR TEST IN 10^{-8} TORR ENVIRONMENT

Foil Location	Approximate Temperature, °F[2]	Analysis, ppm O	N	H
Fourth Layer (Outer)	1220	720	64	10
Third Layer	1540	510	34	8
Second Layer	1780	521	25	8
First Layer (Inner)	1930	465	30	6
Before Test	--	338	28	12

1 Material Control Number 401, 0.002-inch thick by 0.5-inch wide.

2 Temperature of foil calculated using an emittance value of 0.2 and a tube wall temperature of 2065°F.

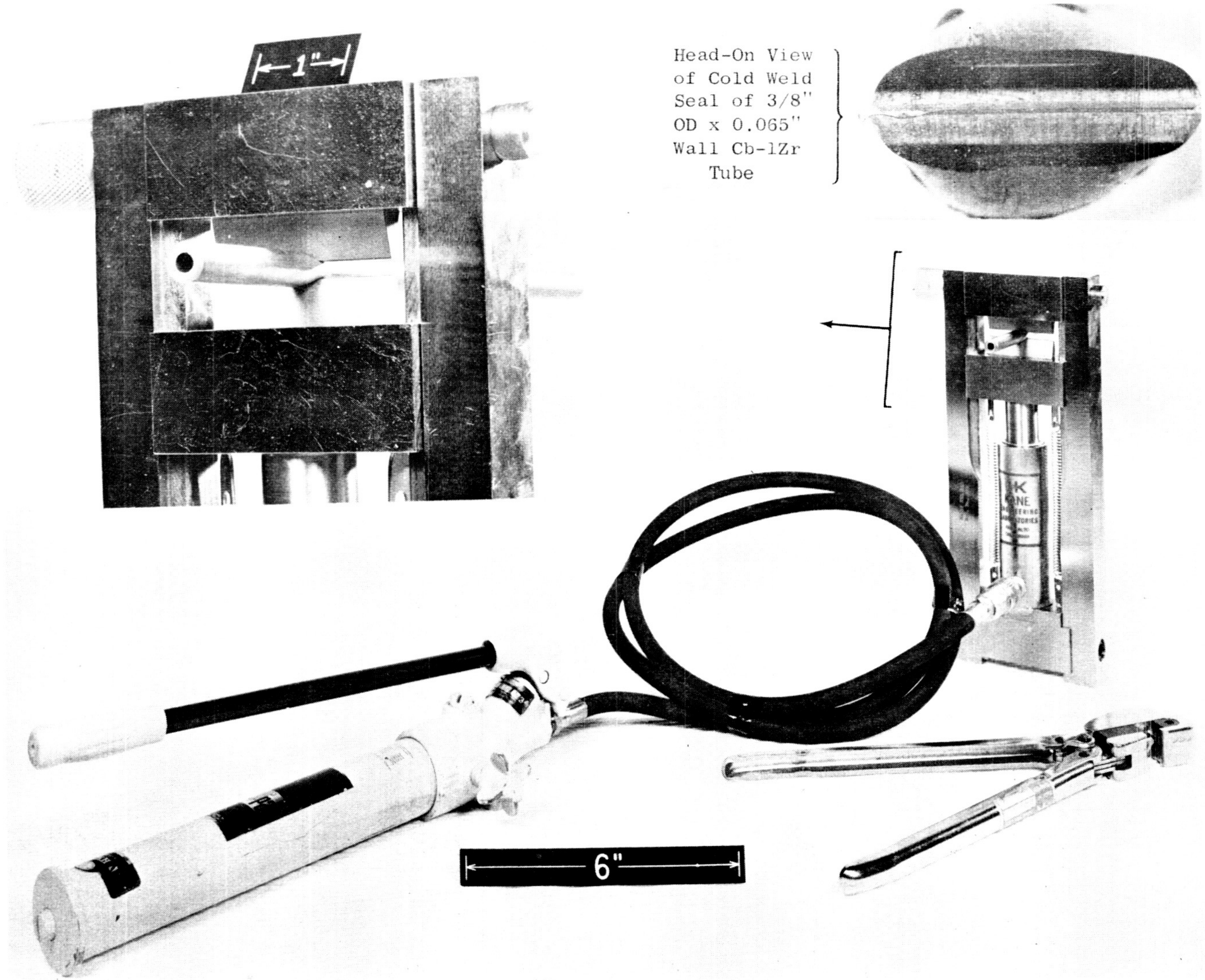


Figure 1. Manual and Hydraulic Cold Weld Pinch-Off Devices Used to Seal Cb-1Zr Alloy Tubing During Sectioning of Corrosion Test Loops.
(Orig. C650621104)

3. Evaluation of the Cb-1Zr Tubing

A total of 25 metallographic specimens, 17 bend specimens, and 13 specimens for chemical analysis were taken from the various regions of the loop.

The locations and temperatures of the various portions of the loop tubing which were analyzed are indicated in Figure 2. The results of these analyses and the before test analysis of the Cb-1Zr tubing are given in Table II. The following conclusions may be made based on these results:

a. In general, very little interstitial contamination occurred during the test, and only the oxygen concentration varied significantly.

b. Analyses obtained on the lowest temperature region (1965°F) and the highest temperature regions (2050° and 2065°F) indicate a transfer of oxygen from the high to the low temperature sections.

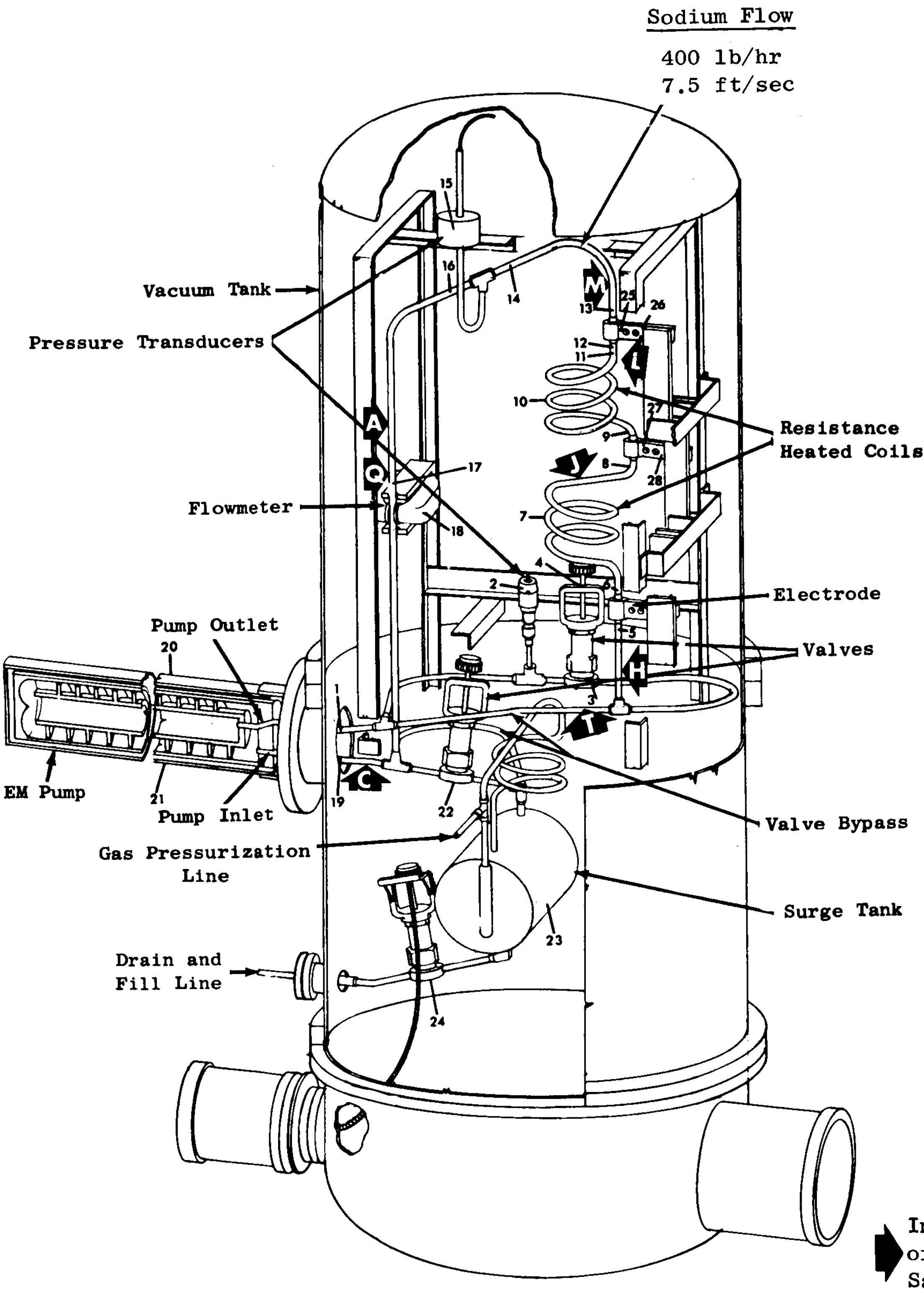
c. Maximum wall contamination (approximately 240 ppm O) occurred in the 2015°F heat rejection area not protected with Cb-1Zr foil wrap.

d. Gradient analyses obtained indicate that all of the oxygen contamination was confined to the outer portions of the tube wall.

The results obtained on Sample M from the 2050°F region of the loop indicate a significant decrease in the oxygen concentration of the Cb-1Zr at the tube-sodium interface in this region. It is of interest to note that the depletion of oxygen from the high temperature regions is in agreement with the depletion noted in the hottest regions of Loop I (4).

Preliminary examination of the metallographic specimens from Loop II has been completed. No corrosion was detected on any of the 21 Cb-1Zr specimens taken from various regions of the loop including one butt weld specimen from the 1965°F heater inlet region. The surface condition of the before test tubing and sections taken from the heater outlet and heat inlet regions is shown in Figure 3. The structures shown are typical of all the specimens of the loop tubing following test.

The post-test ductility of seventeen 1/2-inch long tube sections from various regions of the loop was checked by flattening the tubes using a strip of 0.060-inch thick sheet as an internal support. No cracks could be detected in any of the specimens by visual examinations at 30 diameters. Metallographic examination at 500X revealed several cracks to a depth of 2 to 4 mils on the inner (compression) surface of the bends indicating no significant change in tubing ductility as a result of the 2,650-hour test.



Loop Temperatures

T/C No.	°F
1	1975
2	585
3	800
4	580
5	1965
6	1950
7	1985
8	2020
9	2000
10	2035
11	2065
12	2065
13	2050
14	2045
15	650
16	2030
17	2015
18	670
19	1985
20	1880
21	1850
22	505
23	475
24	455
25	1655
26	1575
27	1720
28	1580

Indicates Location of Chemical Analysis Samples

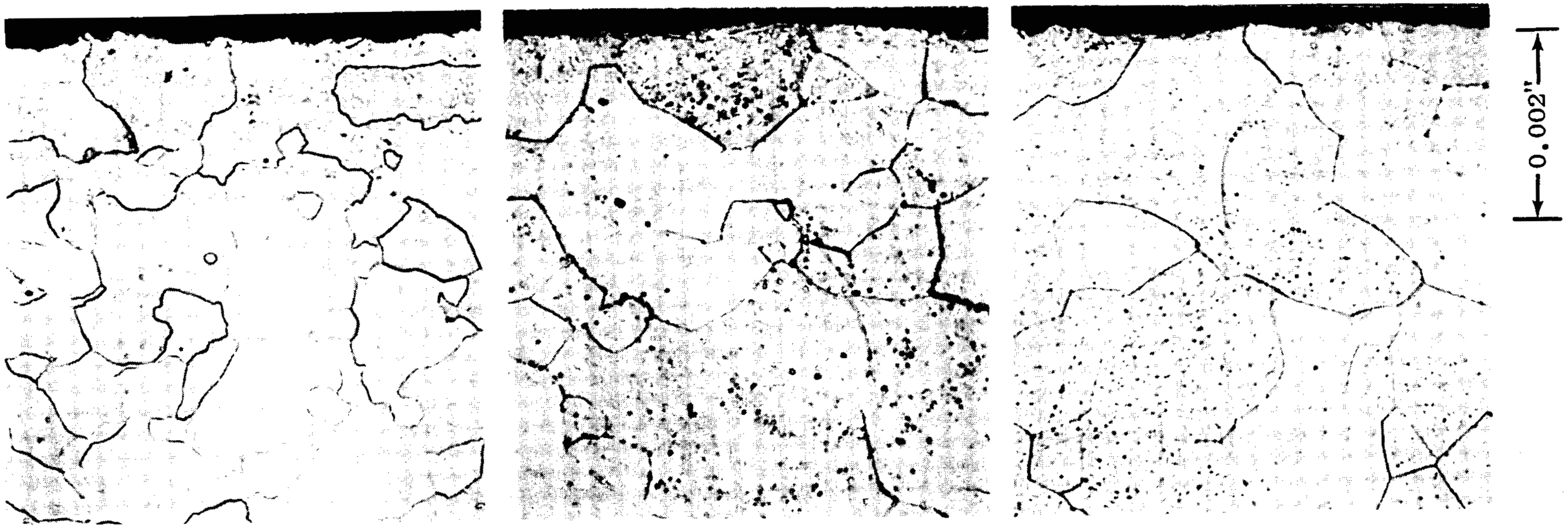
Figure 2. Component Evaluation Test Loop II Showing Various Loop Temperatures During Test Operation with Sodium and the Location of Chemical Analysis Samples.

TABLE II. RESULTS OF CHEMICAL ANALYSIS[1] ON SPECIMENS OF TUBING[2] FROM COMPONENT EVALUATION TEST LOOP II WHICH CIRCULATED SODIUM AT A MAXIMUM TEMPERATURE OF 2065°F FOR 2,650 HOURS

Sample Designation and Description	Chemical Analysis, ppm			
	O	N	H	C
(T) Pump Outlet (1970°F)	254	25	3	-
Inner 1/3 of Wall	350	33	18	-
(H) Heater Inlet (1965°F) - No Foil Wrap	383	58	2	80
(J) End of First Heater Coil (2020°F)	163	42	1	50
(L) End of Second Heater Coil (2065°F)	101	38	2	120
(M) 2 Inches Past Heater Coil (2050°F)	87	41	1	60
Following Removal of 0.010 Inch of Outer Surface of Tube Wall	59	36	3	-
(A) Middle of Cold Leg - No Foil Wrap (2015°F)	404	61	5	110
Outer 1/3 of Wall	569	43	1	80
Middle 1/3 of Wall	188	77	3	100
Inner 1/3 of Wall	146	55	1	80
(Q) Middle of Cold Leg (2015°F)	219	53	4	80
Outer 1/3 of Wall	428	50	6	105
Middle 1/3 of Wall	393	84	4	70
Inner 1/3 of Wall	171	68	3	50
(C) Pump Inlet (1985°F)	207	46	7	60
Before Test Analysis	159	29	5	60

[1] Analysis given is for total wall thickness unless otherwise indicated. All tube specimens covered with Cb-1Zr foil during test unless otherwise indicated. Average of duplicate analyses given in table.

[2] 0.375-inch OD x 0.065-inch wall.



a) Before Test

b) Heater Outlet, 2050°F

c) Heater Inlet, 1965°F

Figure 3. Microstructures of Cb-1Zr Tubing Following 2650 Hours Exposure to Sodium Flowing at 7.5 fps.
Etchant: 60% Glycerine-20%HF-20%HNO_3 Original Mag. 500X
a) B-020111, b) A-090312, c) A-180713

4. Evaluation of Helical Induction EM Pump

Air flow tests of the Loop II sodium pump showed an increase of approximately 3% in the pressure drop over the previous measurement made before the pump was installed in Loop II. The 3% change is probably due to the addition of an inlet and outlet reducer on the pump which was not present in the pretest check. It is, therefore, concluded that no significant changes occurred in the flow characteristics of the pump as a result of the 2,650-hour test near 2000°F. The results are summarized in Table III below:

TABLE III. AIR PRESSURE DROP TEST OF LOOP II SODIUM PUMP

	Pretest	Post-Test[1]
Flow Rate, lbs/hr	7.5	7.5
Pressure Drop, inches of H_2O	32.2	33.3
Pressure Drop Change, %	----	+3

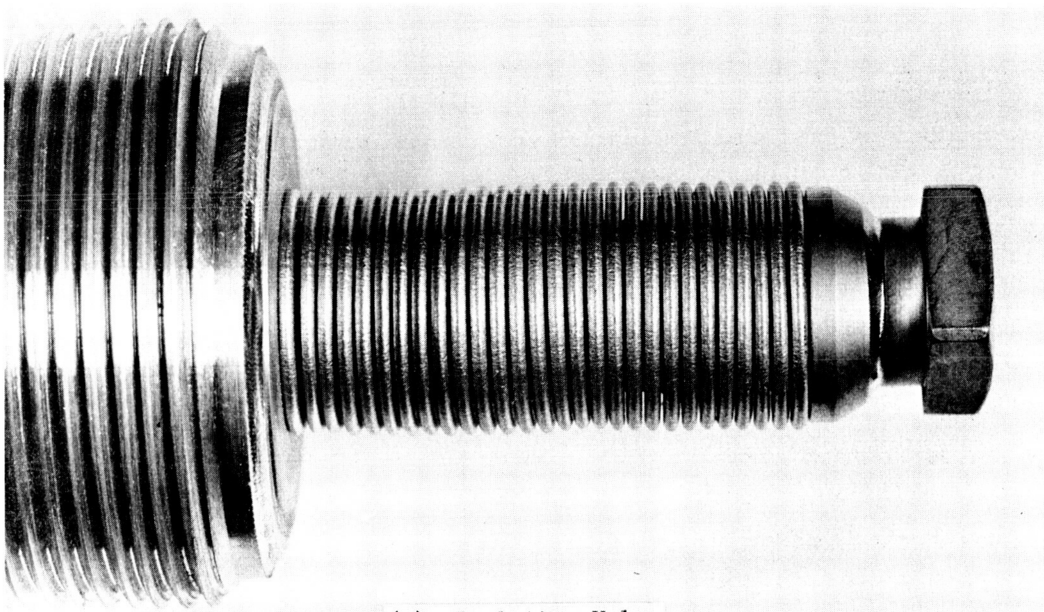
[1] Includes inlet and outlet reducers.

It was also observed that the pressure drop was within 1% for air flow in either direction, i.e., for reverse or forward flow.

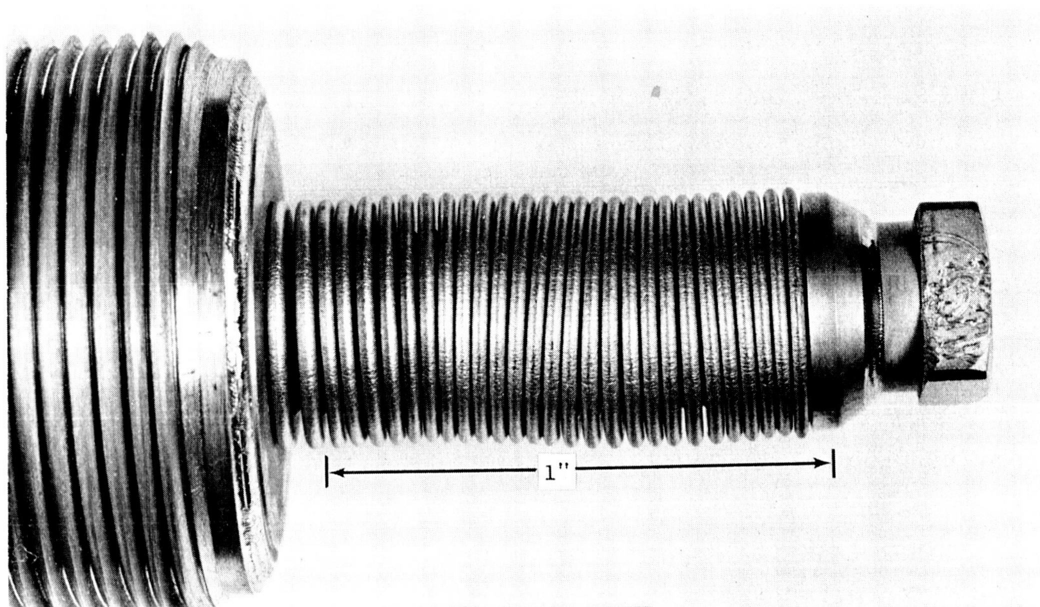
5. Post-Test Appearance of Valve Components

The appearance of the bellows of the isolation and the metering valves from Loop II following completion of the 2,650-hour test is illustrated in Figure 4. Very little difference in the before and after test appearance of the bellows could be detected, however, some evidence of bellows "squirm" in the metering valve bellows is apparent. These valves were operated over the full axial travel range of 0.125 inch approximately 10 times during the pretest checkouts at room temperature. The metering valve was adjusted over an axial movement range of 0 to 0.014 inch approximately 30 times during the 2,650-hour test with the valve at temperatures of 650°-800°F.

Following cleaning of residual sodium from the valve by vacuum distillation, the bellows and plug were removed by cutting out the weld which sealed these parts into the valve body. The bellows and plug are shown



(a) Isolation Valve



(b) Metering Valve

Figure 4. Cb-1Zr Bellows of Modified Hoke Valves Following Completion of Loop II Test. Isolation Valve Bellows (a) Unchanged During Test. Metering Valve Bellows (b) Shows Evidence of Some Distortion.

in Figure 5. No evidence of significant corrosion could be detected, however, the surface of the Mo-TZM alloy plug was covered with a very thin grey film.

6. Recalibration of the Flowmeter Magnet

The sodium flow rate of Loop II was measured by a permanent magnet flowmeter designed to operate at a liquid metal temperature of 2200°F. The magnet had been stabilized by repeated thermal cycling from room temperature to 900°F. The magnetic flux density as a function of temperature after stabilization was measured and reported in a previous quarterly (5). The flux density measurement was repeated after completion of the 2,650-hour Loop II test where the magnet temperature was approximately 670°F during the test operation. In comparing the post-test data with the pretest data, an arithmetical error was detected in computing the pretest flux density resulting in a flux density error of 7%. The corrected pretest flux density and the post-test flux density as a function of temperature are shown in Figure 6. Although no detectable change in the flux density at room temperature was measured, the magnet after test showed a greater sensitivity to temperature than indicated by the pretest measurement. The 2% change in pretest flux density from room temperature to 900°F was still considerably less than the 12 to 13% decrease in flux density obtained for the two Prototype Corrosion Loop magnets of similar design measured over the same temperature range (see III.D.3).

Another anomaly in the behavior of the Loop II magnet is the 10% decrease in the flux density at room temperature following the thermal stabilization treatment. For the two similar Prototype Loop magnets, the flux density after thermal stabilization was only 2% less than the original value.

B. Prototype Corrosion Loop Fabrication

During this report period all the major subassemblies were completed. These subassemblies as defined by the applicable manufacturing instruction are: sodium heater coil, sodium surge tank, sodium EM pump duct, potassium surge tank, potassium EM pump duct - potassium pump outlet, condenser - turbine simulator (stages 2-10), and boiler - turbine simulator (stage 1) preheater coil. The welding and postweld heat treatments required for each of these subassemblies were completed and the final assembly phase was initiated.

The final assembly fabrication phase has been completed through the following operations:

1. Four Cb-1Zr to Type 316 stainless steel bimetallic joints were welded to the surge tank assemblies.

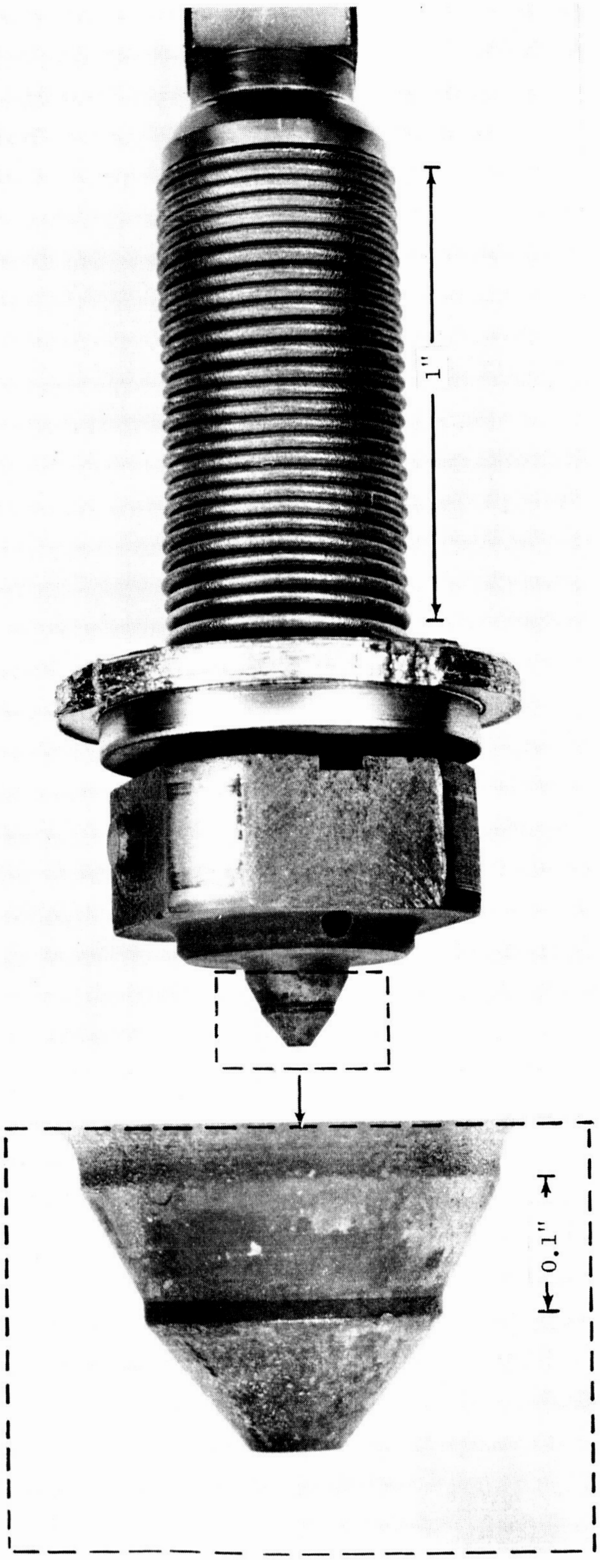


Figure 5. Cb-1Zr Bellows and Mo-TZM Alloy Plug of Loop II Metering Valve Following Disassembly of the Valve.

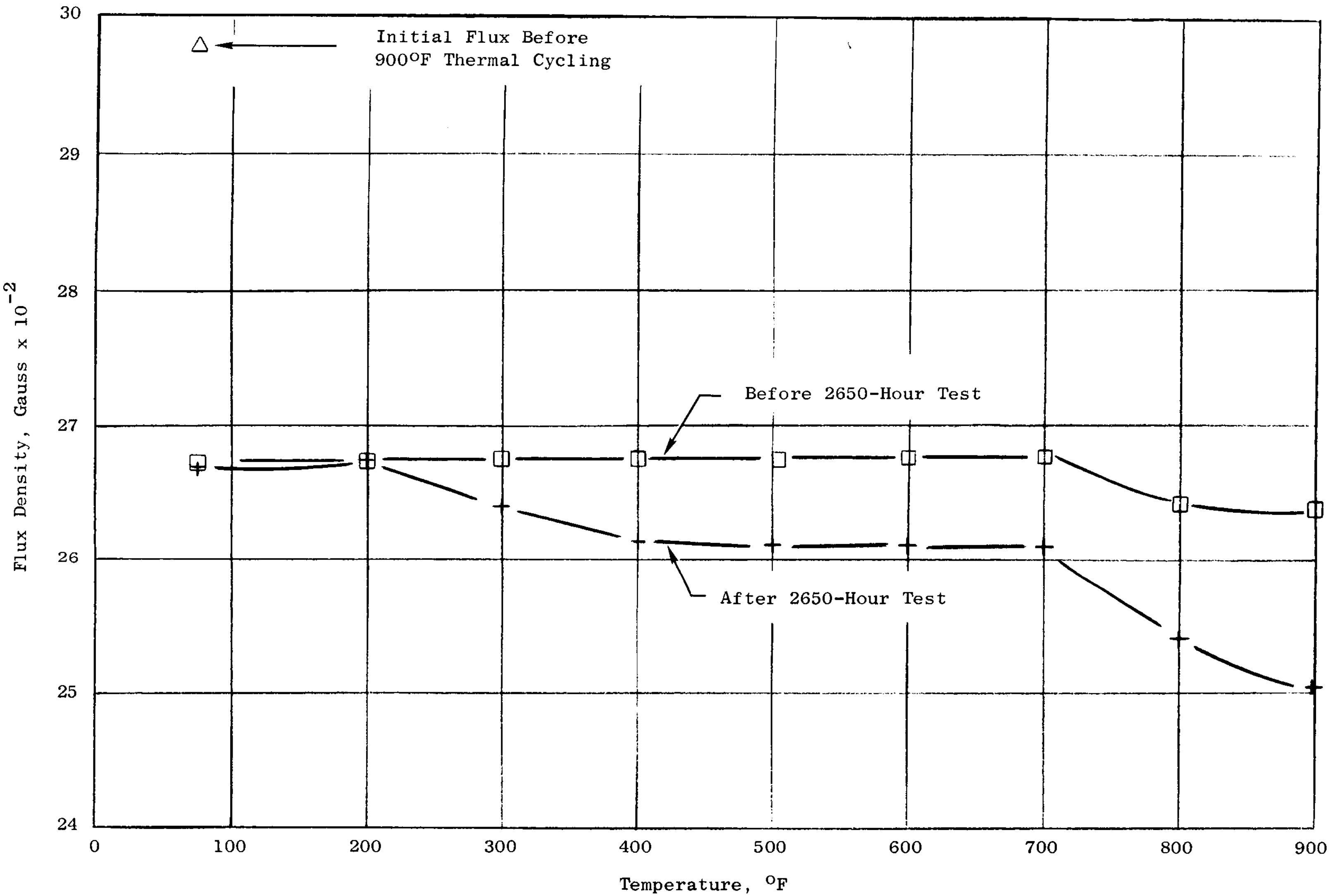


Figure 6. Magnet Flux Density Vs. Temperature of Component Evaluation Test Loop II Flowmeter Magnet Before and After 2650-Hour Test.

2. The sodium system, heater coil, surge tank, and EM pump duct were joined.

3. Components of the potassium system which included the surge tank, EM pump duct, pump outlet, boiler - turbine simulator - preheater coil and two slack diaphragm pressure transducers were joined.

These major subassemblies along with the condenser - turbine simulator part of the potassium system are currently being positioned on the support structure and vacuum tank spool section. When this step is completed, the loop welding fixture will be positioned inside the support frame and the attachments to the loop will be made. The loop support structure will then be disassembled and the loop, supported by its welding fixture, will be positioned in the welding chamber.

A total of seven welds and local postweld annealing heat treatments will then be required to complete the Cb-1Zr portion of the loop assembly.

After reassembly in the vacuum tank spool section, the Type 316 stainless steel fill and dump lines, and gas pressure lines will be welded to the vacuum chamber ports. Also, stainless steel components of the housings for the EM pumps and of the vacuum feedthroughs for the slack diaphragm transducers will be welded in place.

The fabrication sequence for loop components and major subassemblies is described below:

1. Boiler

The boiler fabrication through initial welding and forming operations has been reported previously (6). During this report period, welding of the boiler components shown in Figure 7 was completed. The inner and outer tubes were cut to the proper length, and the tube plug was welded to the inner tube. After inspection of this weld, the end connectors for the outer tube were positioned and welded along with the thermocouple wells and inlet plug. Radiographs of the potassium inlet section shown in Figure 8 illustrate the inlet plug configuration. A 0.060-inch diameter Cb-1Zr wire was formed into a 1.0-inch pitch helix on the 0.125-inch diameter Cb-1Zr center rod. One TIG weld tack per revolution of the plug wire was made to hold the wire in place. The plug was then positioned within the straight section of boiler inlet pipe and welded to the inlet fitting. The welded assembly x-ray and leak test was inspected prior to its inclusion in a major subassembly described below.

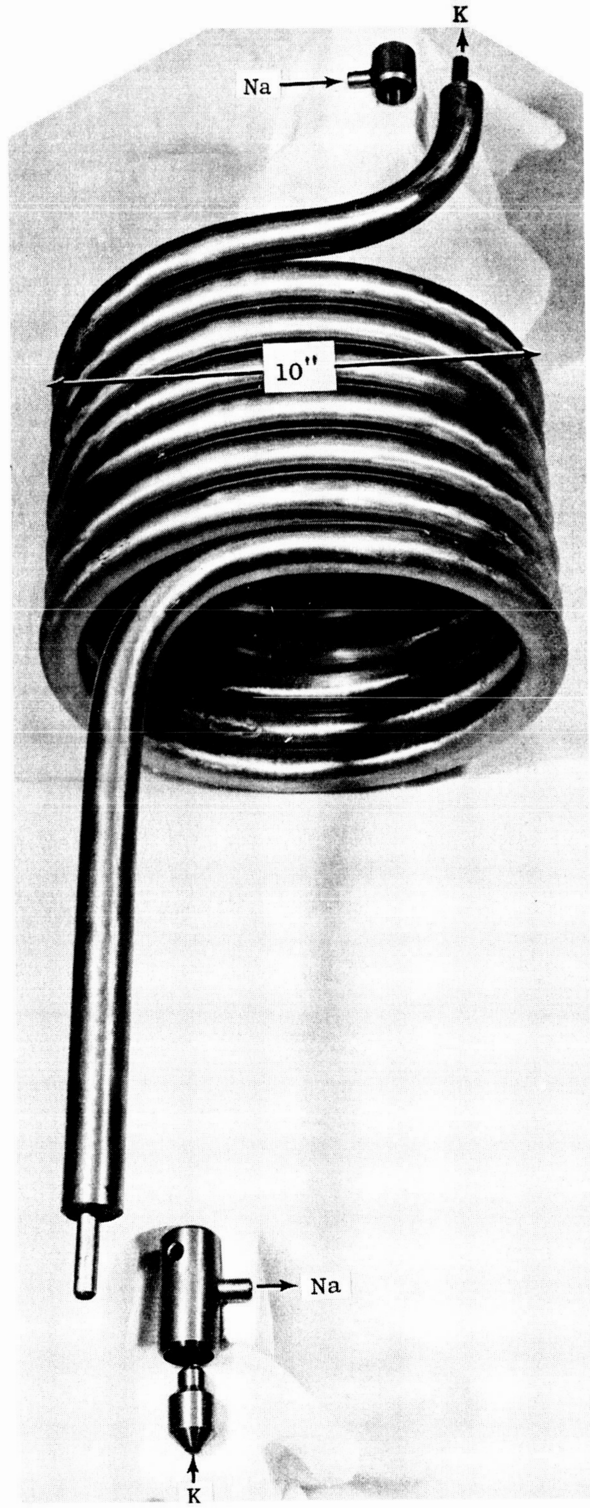


Figure 7. Prototype Corrosion Loop Tube-in-Tube Helical Boiler. C650621101

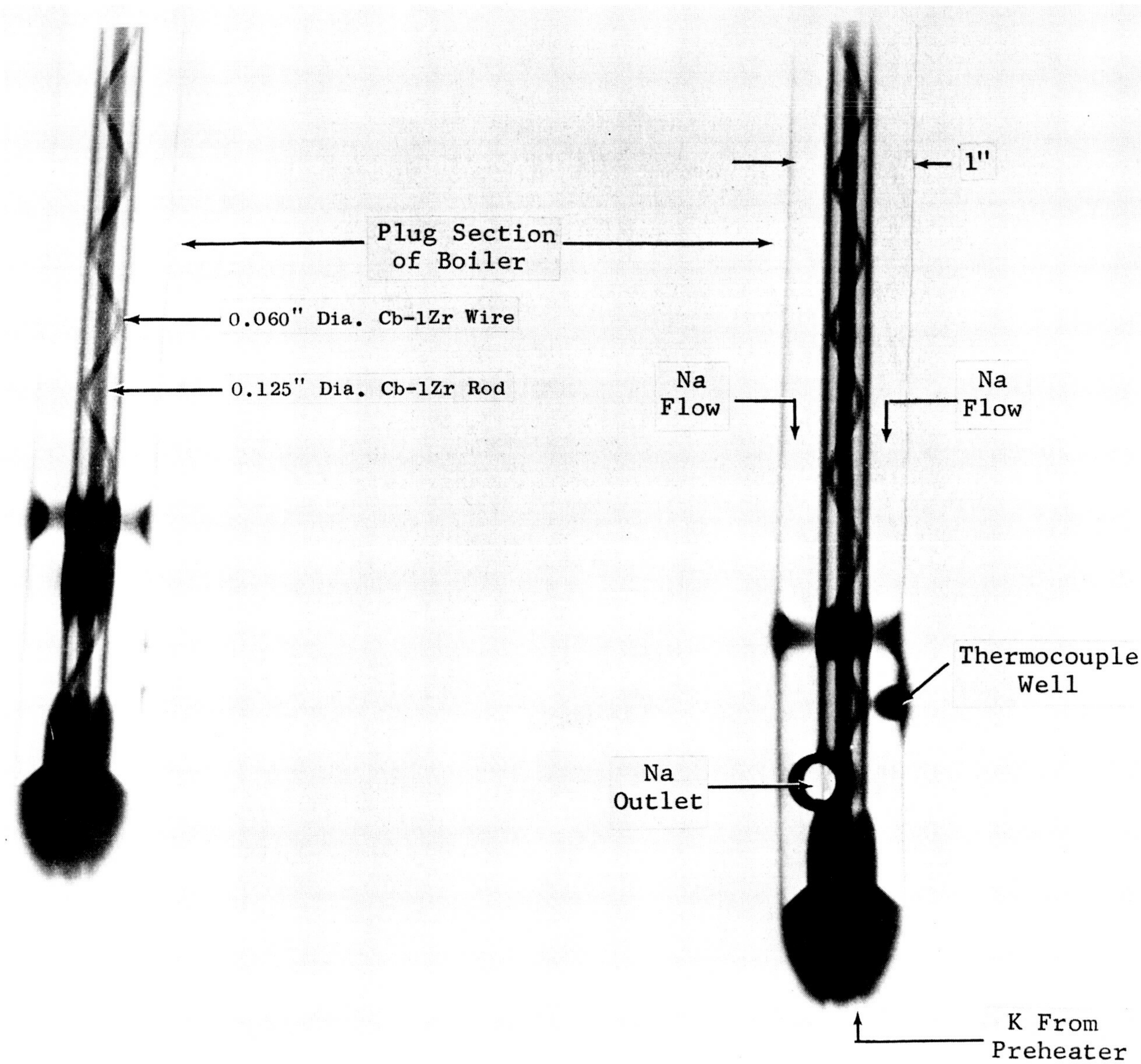


Figure 8. Two Radiographs of the Boiler Inlet Region of the Prototype Corrosion Loop Showing the Plug Configuration.

2. Turbine Simulator

The machining and assembly of the turbine simulator nozzles and blades was completed. The tenth stage nozzle configuration is shown in Figure 9. The Mo-TZM alloy blades were polished to a 16-rms surface finish by the vendor utilizing a 240 grit alumina cloth followed by a final polish with 5-micron alumina before final cleaning and assembly. The Mo-TZM alloy nozzles were Elo-polished, a refinement of electric spark discharge machining, to produce a 32-rms surface finish in the nozzle throat. This surface was further polished with 400 grit alumina paper to remove approximately 0.001 inch from the surface. A final polish with 5-micron alumina produced an 8-rms finish. Zyglo inspection of the finished parts indicated that the surfaces were free of cracks. It should be noted that numerous surface cracks were detected in the blade pads (see Figure 9) after electric spark discharge machining of the airfoil shape. The majority of the cracks were located in the sharp radii at each end of the airfoil shape and were easily removed by filing and/or hand polishing with 5-micron alumina powder as required.

Each nozzle orifice diameter was air-gauge inspected for final dimensions with the results shown in Table IV. Nozzle number 9 was 0.0008 inch oversize, number 2 was 0.0001 inch oversize, and number 4 was 0.00003 inch oversize in one location. These minor discrepancies will not influence loop performance.

The nozzle assemblies were then disassembled, cleaned with ethyl alcohol, and the weights of each component were recorded for future reference.

The number one nozzle assembly was positioned in its Cb-1Zr outer casing as shown in Figure 10. The end caps, thermocouple wells, and one-inch diameter process tube were then welded to complete the single stage turbine simulator. Postweld annealing of this subassembly was conducted after it was joined to the boiler to form a major subassembly.

The remaining nine nozzle assemblies were positioned as shown in Figure 11. These assemblies are rabbeted together and aligned in their Cb-1Zr casing with a 0.062-inch diameter wire which extends through a keyway in the nozzles and casing. Their assembly in the casing was somewhat complex since both unit straightness and axial alignment had to be maintained. It was found that additional diametral clearance between the nozzles and casing was required during assembly. This additional clearance was obtained by heating the Cb-1Zr casing to approximately 300°-350°F in air. Using this procedure, the delicate fitting operation was accomplished. The assembly was at the temperature specified for approximately 15 minutes. The Cb-1Zr end caps and thermocouple wells were then welded to the casing to complete the assembly.

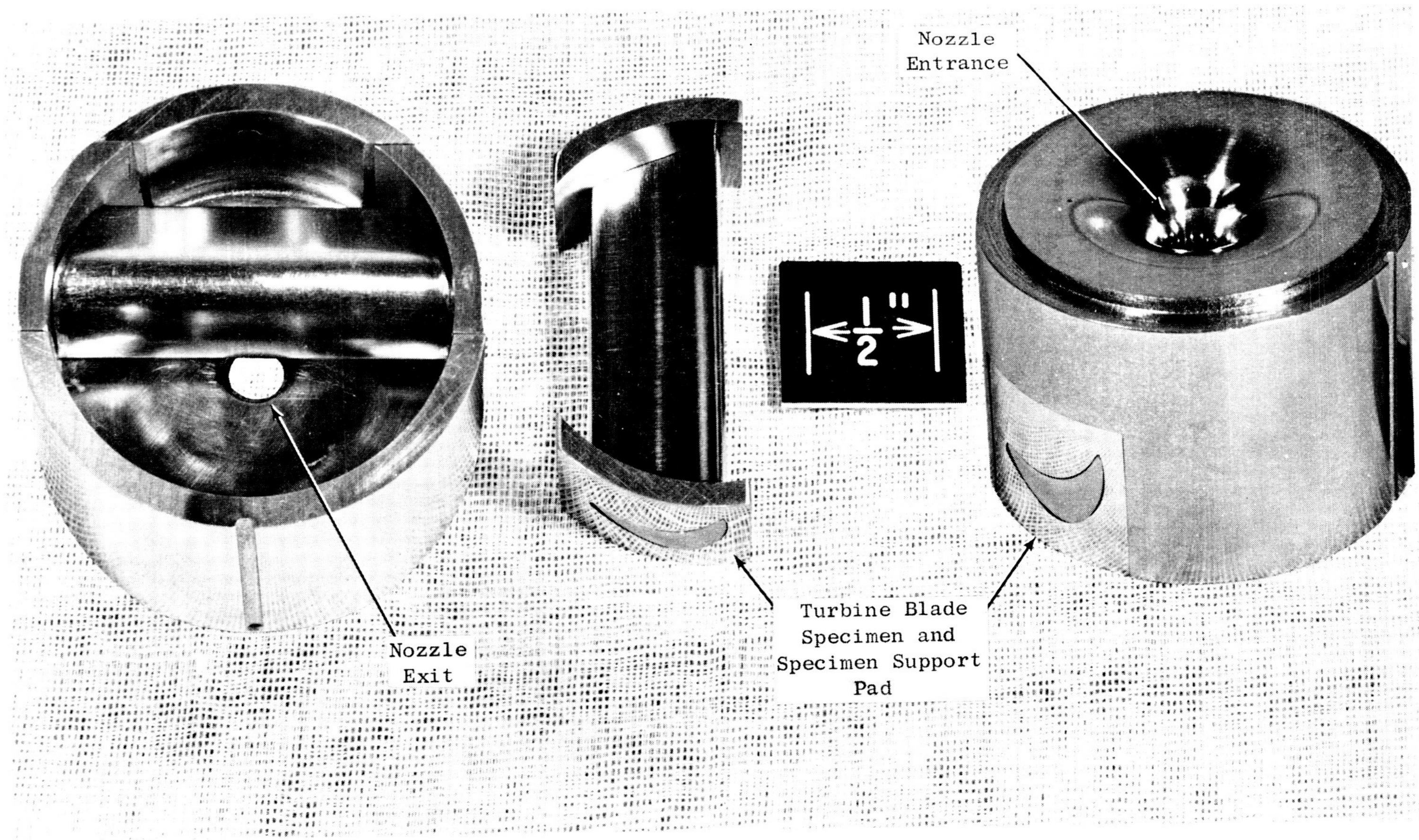


Figure 9. Mo-TZM Alloy Turbine Simulator Nozzle and Blade Assembly.

TABLE IV. ORIFICE DIAMETERS FOR THE Mo-TZM ALLOY NOZZLES FOR THE PROTOTYPE CORROSION LOOP TURBINE SIMULATOR AS DETERMINED BY AIR-GAUGE INSPECTION

Stage No.	Identification	Specified Diameter, Inches	Actual Diameter, Inches	
			0 Degrees[1]	90 Degrees
1	Dwg. 119C2853 P1	0.099 - 0.100	0.09940	0.09945
2	Dwg. 119C2853 P2	0.100 - 0.101	0.101080	0.101100
3	Dwg. 119C2853 P3	0.115 - 0.116	0.115300	0.115270
4	Dwg. 119C2853 P4	0.128 - 0.129	0.129030	0.128960
5	Dwg. 119C2853 P5	0.141 - 0.142	0.141170	0.141205
6	Dwg. 119C2853 P6	0.158 - 0.159	0.158050	0.158025
7	Dwg. 119C2853 P7	0.175 - 0.176	0.175720	0.175660
8	Dwg. 119C2853 P8	0.205 - 0.206	0.205080	0.205075
9	Dwg. 119C2853 P9	0.221 - 0.222	0.222790	0.222785
10	Dwg. 119C2853 P10	0.249 - 0.250	0.249225	0.249215

[1] In line with OD slot on Stages No. 2 through 10.

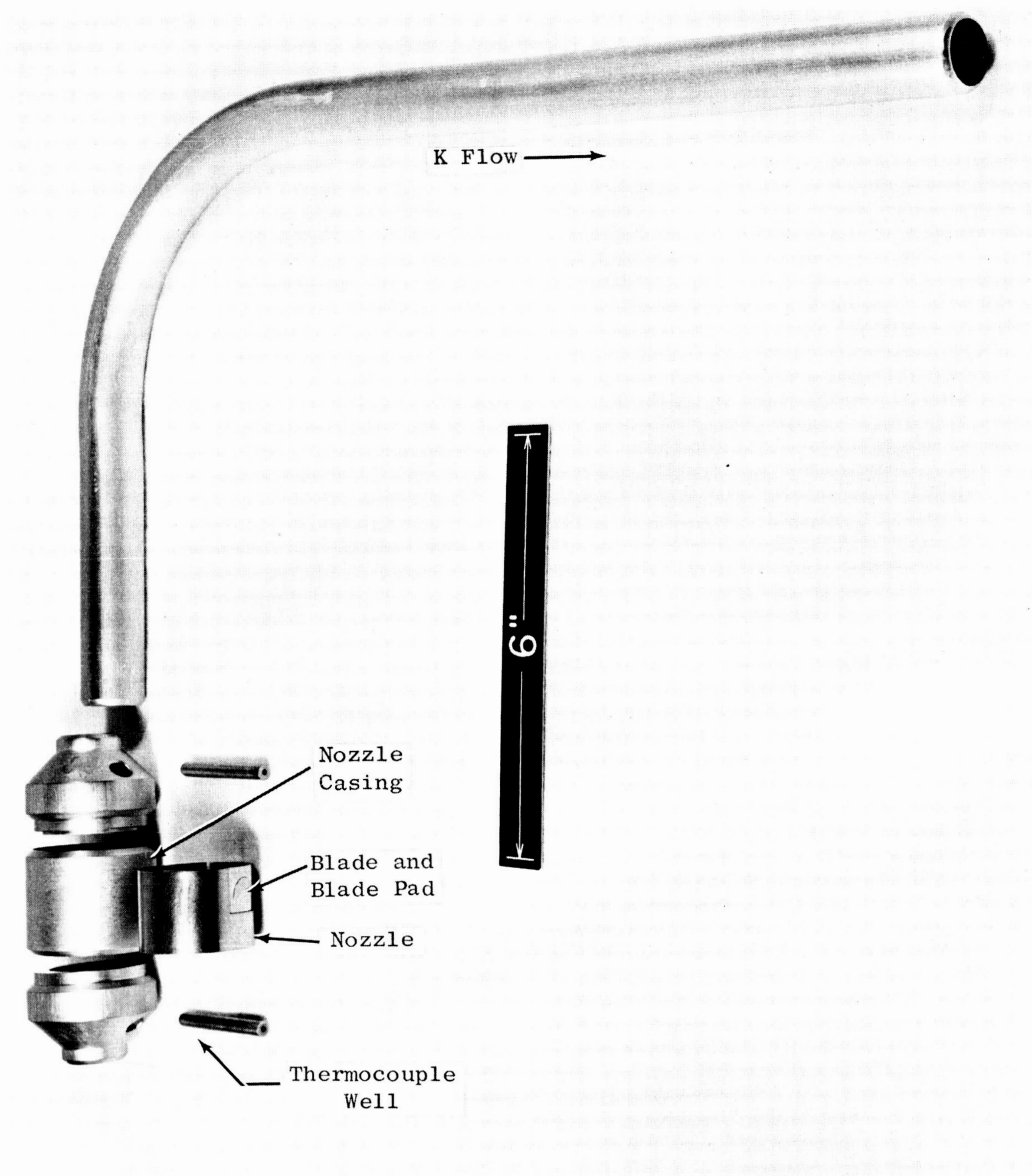


Figure 10. First Stage Turbine Simulator of Prototype Corrosion Loop.
(Orig. C65030136)

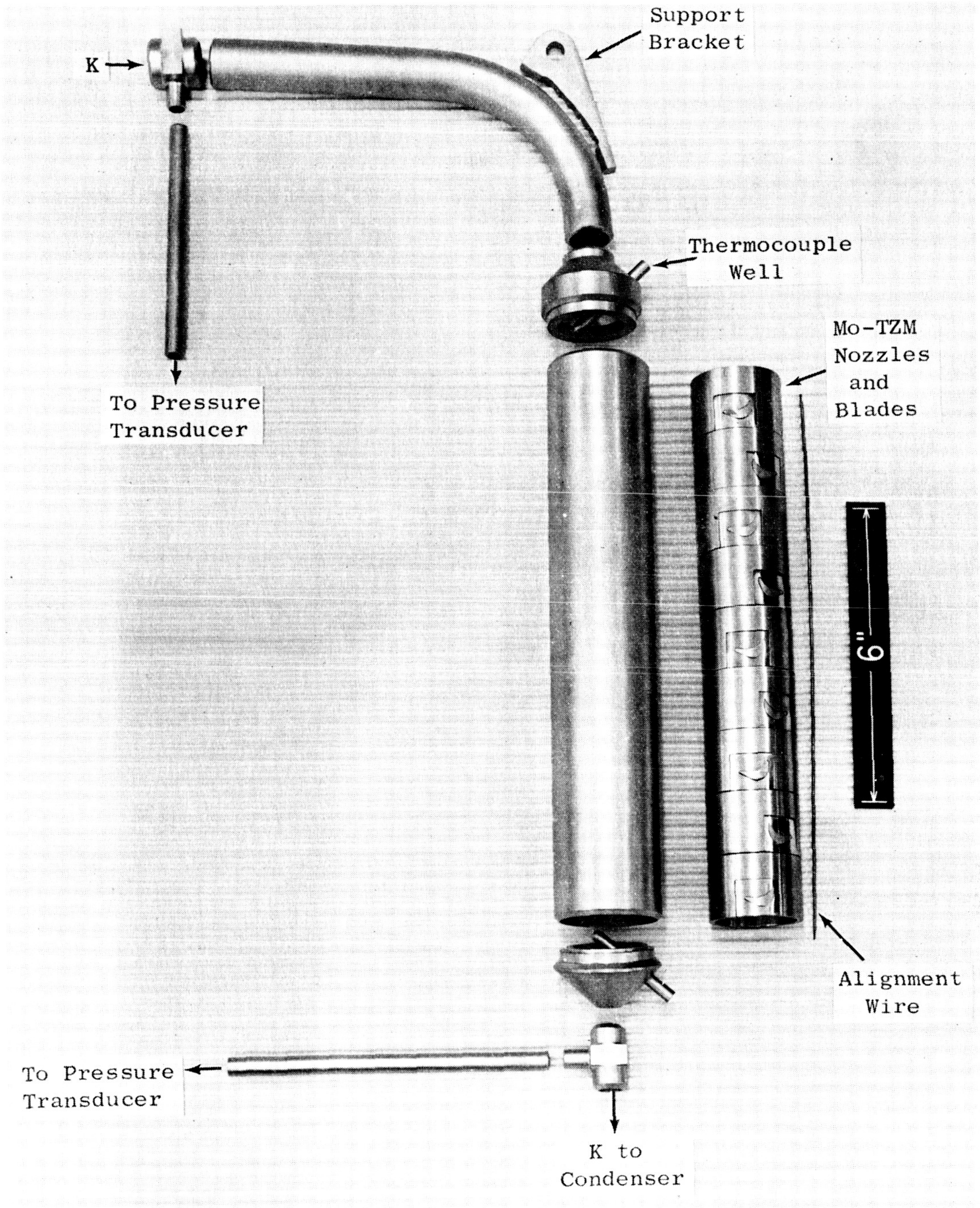


Figure 11. Turbine Simulator (Stages 2-10) of the Prototype Corrosion Loop Prior to Assembly. Nozzles and Blades: Mo-TZM. Tubing, Reducers, Casing and Miscellaneous Fittings: Cb-1Zr.

C65062198

3. Helical Induction EM Pump Ducts

The primary and secondary EM pump ducts were postweld vacuum annealed at Stellite Division of Union Carbide Company. As in previous instances, it was found necessary to deviate from Specification SPPS-3C by setting the upper limit of pressure near 5×10^{-5} torr rather than the 1×10^{-5} torr specified. This upper limit of pressure was considered acceptable since qualification runs which preceded the actual annealing of the loop components yielded acceptable results at the higher pressure. As an added precaution, one over-lapping layer of 0.002-inch thick Cb-1Zr foil was used to wrap the EM pump ducts. In accordance with Specification SPPS-3C, the components were annealed at 2200°F for one hour. The vacuum maintained at 2200°F was 6.5×10^{-5} torr. As shown in Table V, no contamination of control specimens was evident. The completed EM pump ducts are shown in Figure 12.

4. Sodium and Potassium Surge Tanks

Both the potassium and sodium surge tank subassemblies were welded and inspected. These subassemblies include the argon gas pressure line, fill and dump line, tank outlet line, and surge tank as illustrated by the potassium surge tank shown in Figure 13. As in previous instances, it was found necessary to deviate from Specification SPPS-3C by setting the upper limit of pressure near 5×10^{-5} torr rather than the 1×10^{-5} torr specified. In this case, the vacuum maintained during the one hour at 2200°F was 7.5×10^{-5} torr. As shown in Table V, no contamination of control specimens was evident. As in previous instances, one overlapping layer of 0.002-inch thick Cb-1Zr foil was used to wrap the surge tanks and associated tubing.

5. Valves

The fabrication sequence for the Cb-1Zr bellows valves for both Loop II and the Prototype Loop was reported previously (7). During Loop II operation (8), the valve actuation system was further modified by inclusion of a Saginaw ball bearing screw to eliminate galling tendencies. For the Prototype Loop, additional design and materials modifications were made and these have been incorporated in the valve shown in Figure 14. All components which may operate above the 600°F ambient temperature were made from Mo-TZM or Cb-1Zr alloy. Also, a tungsten carbide bushing was added to minimize galling of the stainless steel pinion gear shaft.

6. Stress Diaphragm Pressure Transducer

The design of stressed diaphragm pressure transducer was modified from that used in Loop II by the inclusion of a corrugated 0.006-inch thick T-222 tantalum alloy diaphragm. The transducer is shown in Figure 15 at various stages

TABLE V. RESULTS OF CHEMICAL ANALYSES OF Cb-1Zr ALLOY WELDS[1] AFTER HEAT TREATMENT

	Pressure, Torr		Element, ppm			
	Start of Run	End of Run	O	N	H	C
Base Metal Analysis by Vendor (MCN 454)[2]			90	42	5	35
Base Metal Analysis by GE (MCN 454)			56	14	6	10
Weld Metal-As Welded			69	10	1	10
Weld Metal After 2200°F/One Hour in C.I. Hayes, Inc. Furnace (Model PS-107, 20-Inch Diameter x 36-Inch) at Stellite Division, UCC (Wrapped)[3] - Sample Which Accompanied EM Pump	6.8×10^{-5}	6.5×10^{-5}	65	23	1	10-50
Weld Metal After 2200°F/One Hour in C.I. Hayes, Inc. Furnace (Model PS-107, 20-Inch Diameter x 36-Inch) at Stellite Division, UCC (Wrapped)[3] - Sample Which Accompanied Surge Tank Assemblies	7.5×10^{-5}	7.5×10^{-5}	79	21	2	35

1 Only weld metal portion of weldments analyzed.

2 0.040-inch thick sheet.

3 Specimens and components wrapped with one overlapped layer of 0.002 inch Cb-1Zr foil.

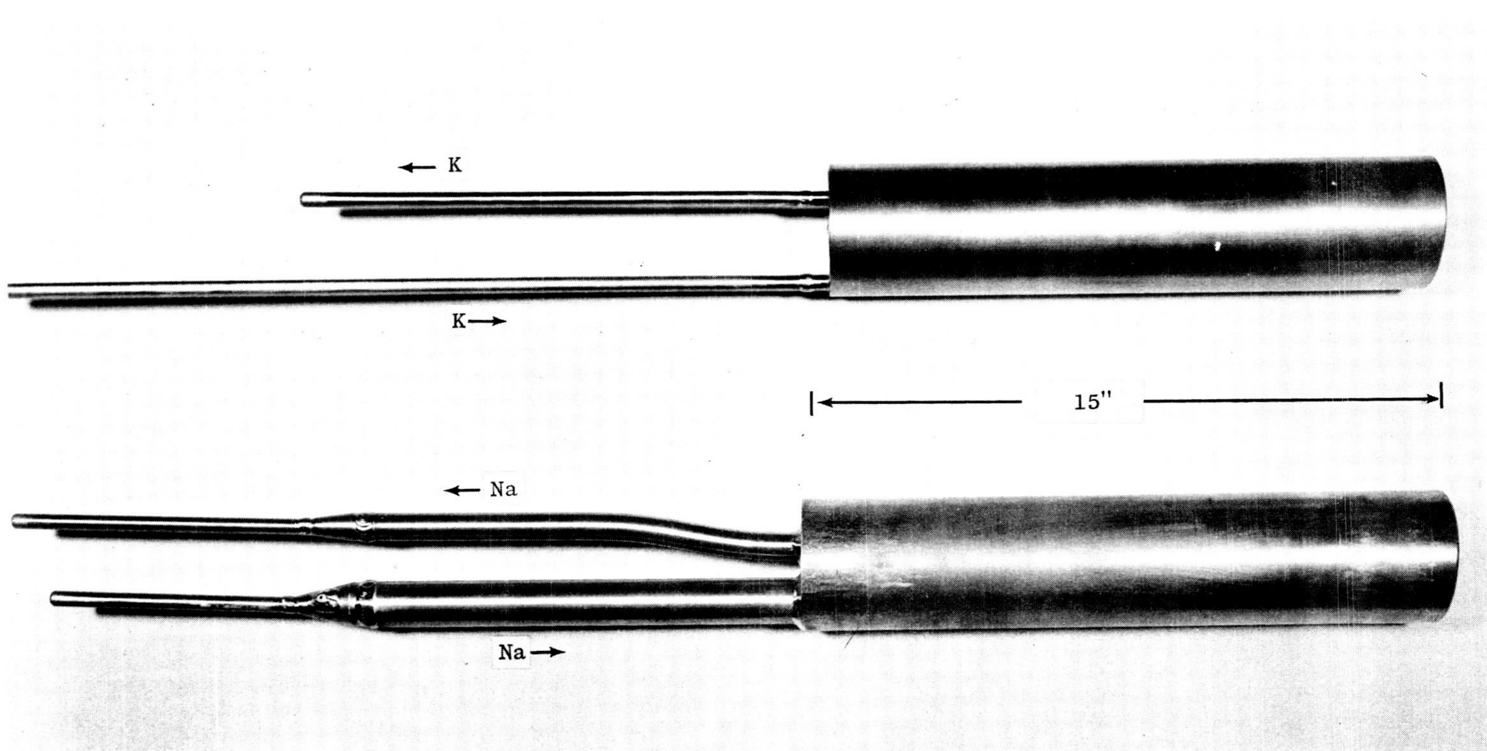


Figure 12. Potassium Duct (Top) and Sodium Duct (Bottom) of the Helical Induction Electromagnetic Pumps for the Prototype Corrosion Loop. All Parts Made of Cb-1Zr Alloy. (Orig. C65012829)

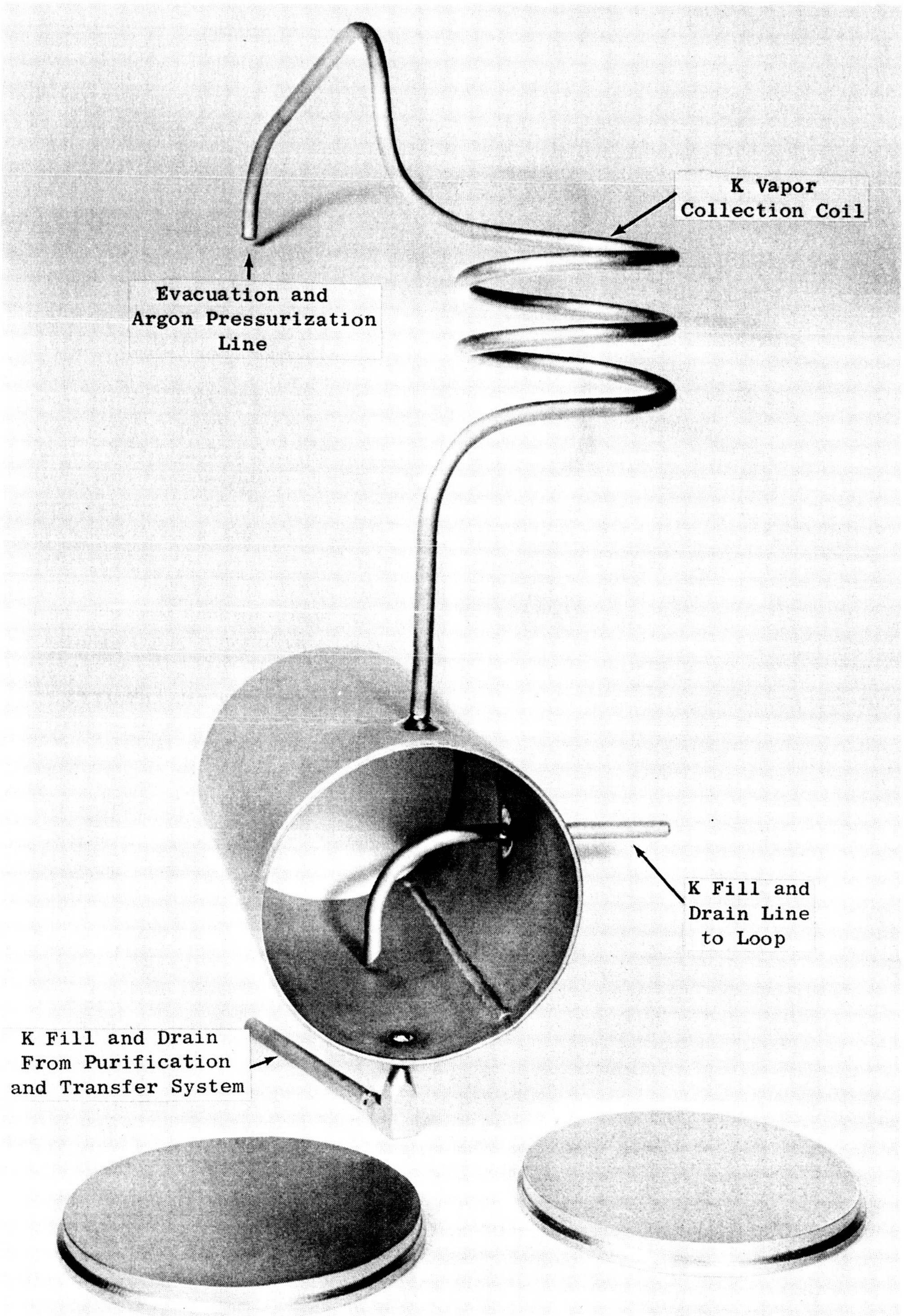


Figure 13. Potassium Surge Tank (6" OD) for the Prototype Corrosion Loop Partially Assembled. (Orig. C65021044)

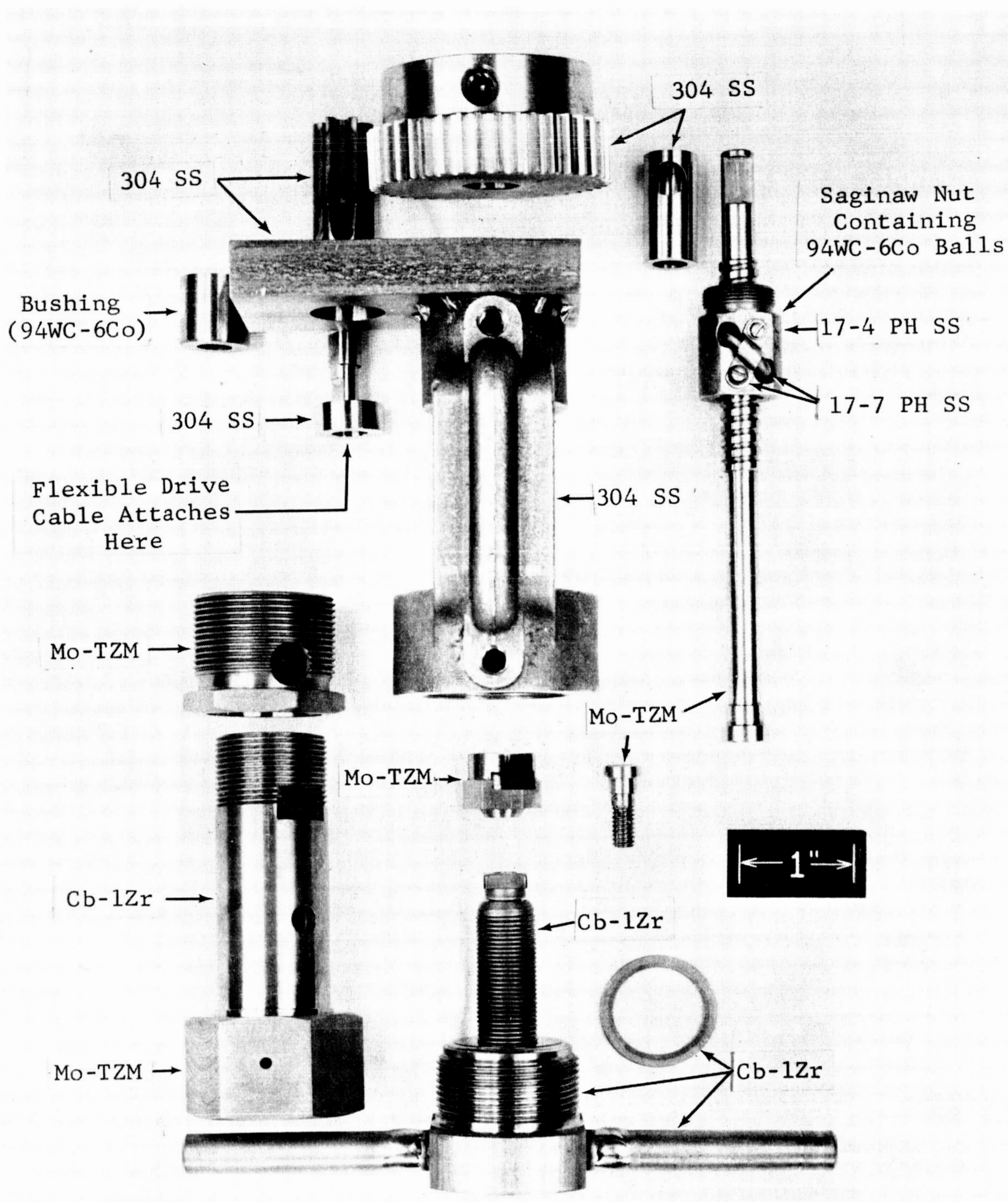
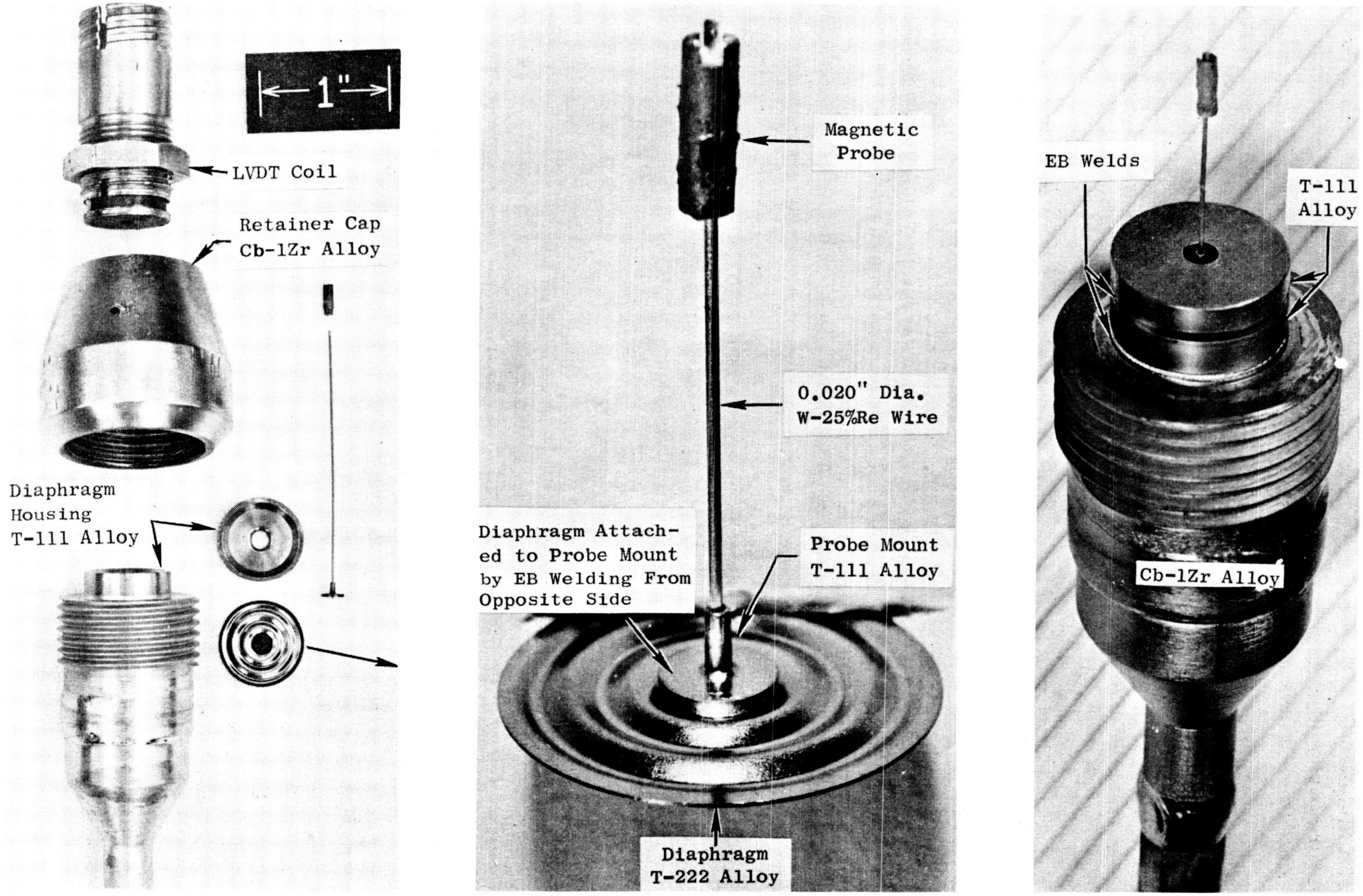


Figure 14. Modified Hoke Valve and Associated Drive Components for the Prototype Corrosion Loop. (C65082449)



a) b) c)

Figure 15. Stressed Diaphragm Pressure Transducer for the Prototype Corrosion Loop at Various Stages During Fabrication. (C65062193)

during the fabrication sequence. Since the flat diaphragm used in the earlier design and the new corrugated diaphragm had different diameters, the transducer housing and housing cap were re-machined as shown in Figure 15(a).

As reported previously (9), the transducer body assembly was welded and postweld annealed per Specification SPPS-3C. The T-111 tantalum alloy housing was first welded to the Cb-1Zr retainer body which was then welded to the reducer and process tube. This subassembly was then annealed at 2200°F for one hour.

The diaphragm assembly shown in Figure 15(b) was produced by first electron beam welding the 0.020-inch diameter W-25%Re wire to the T-111 alloy probe mount. A second electron beam weld attached the probe mount to the T-222 alloy diaphragm. The diaphragm was then positioned between the T-111 housing and housing cap and electron beam welded. As an added precaution, an electron beam weld was made between the T-111 alloy housing and the Cb-1Zr retainer body on the outside diameter, which, in combination with the internal tungsten inert gas weld, affected a double seal between these components.

The final fabrication step was the brazing of the magnetic probe to the 0.020-inch diameter W-25%Re wire. Brazing was accomplished using localized heating with a graphite-tipped heater probe under high purity argon in the welding chamber. The brazing alloy (72Ag-28Cu) was applied to the joint manually in wire form. The completed assembly, shown in Figure 15(c), was mass spectrometer leak tested successfully and submitted to instrumentation for room temperature calibration.

7. Condenser - Turbine Simulator Subassembly

The weld between the condenser pipe and the 0.25-inch thick radiator fins represented the largest continuous weldment required for loop fabrication. Approximately 1.5 pounds of 0.125-inch diameter filler wire was consumed. Alignment of the fins and condenser pipe was maintained by a welding fixture shown in Figure 16. All the welding fixture parts which contact the Cb-1Zr were made from unalloyed molybdenum.

As expected from earlier welding trials, the 0.375-inch OD Schedule 80 condenser pipe was distorted by the welding operation. Radiographic inspection of the condenser assembly revealed a decrease in inside diameter in a direction parallel to the fins. The nominal 0.421-inch inside diameter in this direction decreased to 0.295-0.315 inch. At each of the 0.050-inch gaps between the fins, weld shrinkage was less pronounced with the inside diameter decreasing to a minimum of 0.370-0.380 inch. On an axis perpendicular to the fins, the inside diameter increased to 0.425-0.440 inch.

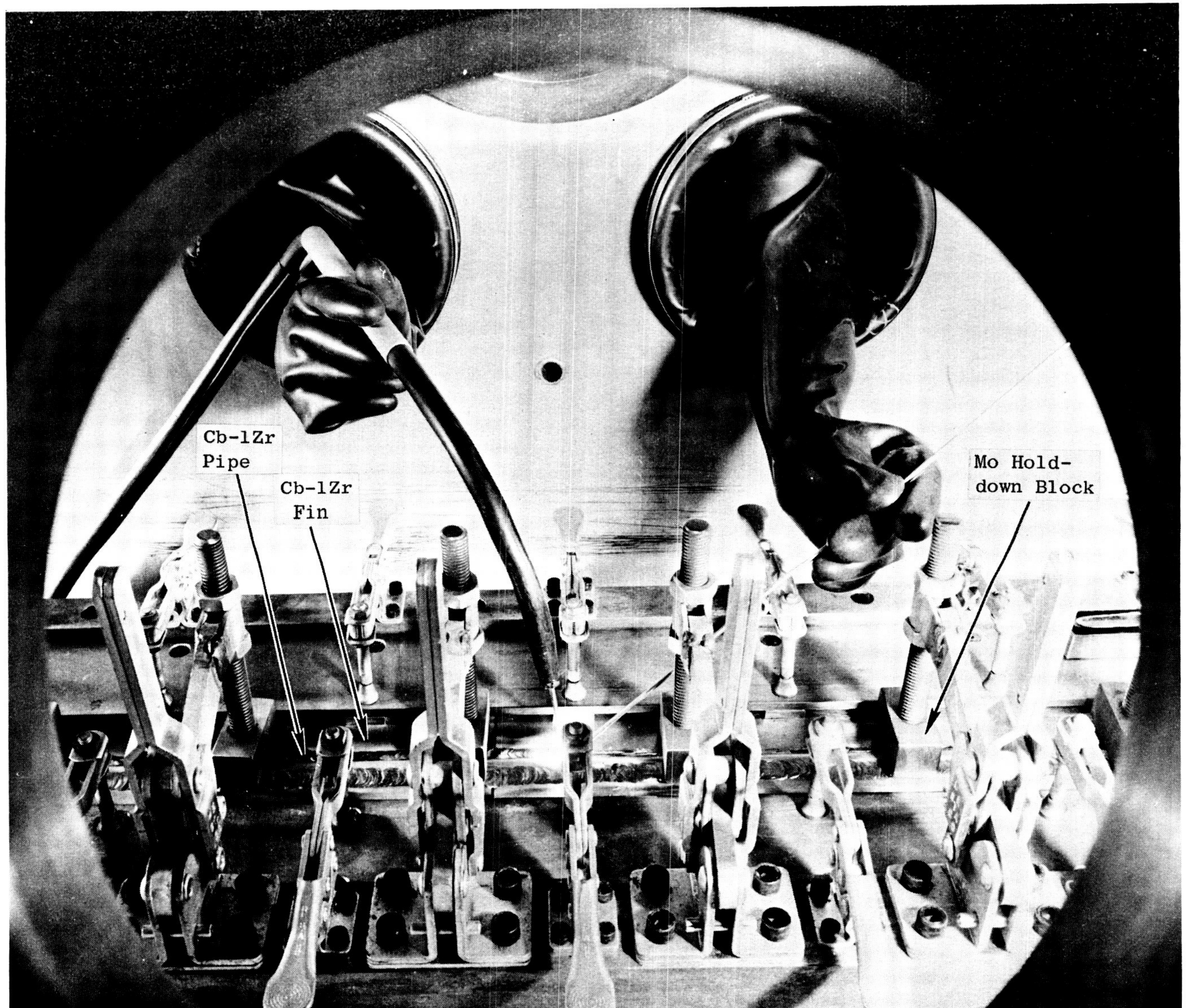


Figure 16. Prototype Corrosion Loop Condenser and Fixture During Welding of Fin to Condenser Pipe. (Orig. C65022602)

Following attachment of the fins, the condenser was then welded to the subcooler and the nine-stage turbine simulator which completed this major subassembly as shown in Figure 17(a). This subassembly was then wrapped with 0.002-inch Cb-1Zr foil in preparation for postweld annealing treatment.

8. Boiler - Turbine Simulator - Preheater Coil Subassembly

This major subassembly which constitutes about one half of the potassium loop was completed as shown in Figure 18. Four welds were required to join the components which included the preheater coil, vapor nucleator, boiler, transducer connector, single stage turbine simulator and 1.0-inch diameter tube. This major subassembly was then wrapped with 0.002 inch Cb-1Zr foil in preparation for the postweld annealing treatment described below.

This subassembly is also representative of the procedure utilized for joining the 0.375-inch diameter process tubes to the larger diameter piping sections. As shown in Figure 18, short lengths of the 0.375-inch tubing were welded to the transducer connectors, boiler inlet and outlet, and preheater electrode prior to postweld annealing treatment. This procedure permitted final assembly welds and local postweld annealing treatments to be performed on the 0.375-inch tubing at readily accessible locations. Only one weld in the 1.0-inch diameter tube at the top of the loop will be required during the final assembly phase.

9. Postweld Annealing Treatments

As noted in the last quarterly report (10) six welded test samples were annealed in the DuPont Company vacuum furnace for two hours at 2200°F $\pm$ 50°F in preparation for the postweld annealing of major loop subassemblies.

The condenser - turbine simulator, Figure 17(a), the boiler - turbine simulator preheater coil, Figure 18, and the sodium heater coil subassemblies were postweld annealed during one furnace run. The application of the high emittance coating to the condenser, which is described in the next section of this report, was done following annealing of this component. Quality assurance specimens included with the furnace run were 1.0-inch and 0.375-inch diameter tubes, and two specimens of 0.040-inch thick Cb-1Zr sheet.

After positioning of the three subassemblies, the furnace was evacuated overnight with hot water circulating through the cooling channels to promote outgassing. After turning on the cold water, furnace vacuum was 1×10^{-6} torr. At this point, furnace power was turned on and the components were heated to 600°F for two hours. During the first one-half hour, the furnace pressure increased to 2.5×10^{-4} torr then decreased to 8×10^{-6} torr at the end of two hours at 600°F. A gradual temperature increase to 2200°F was accomplished in

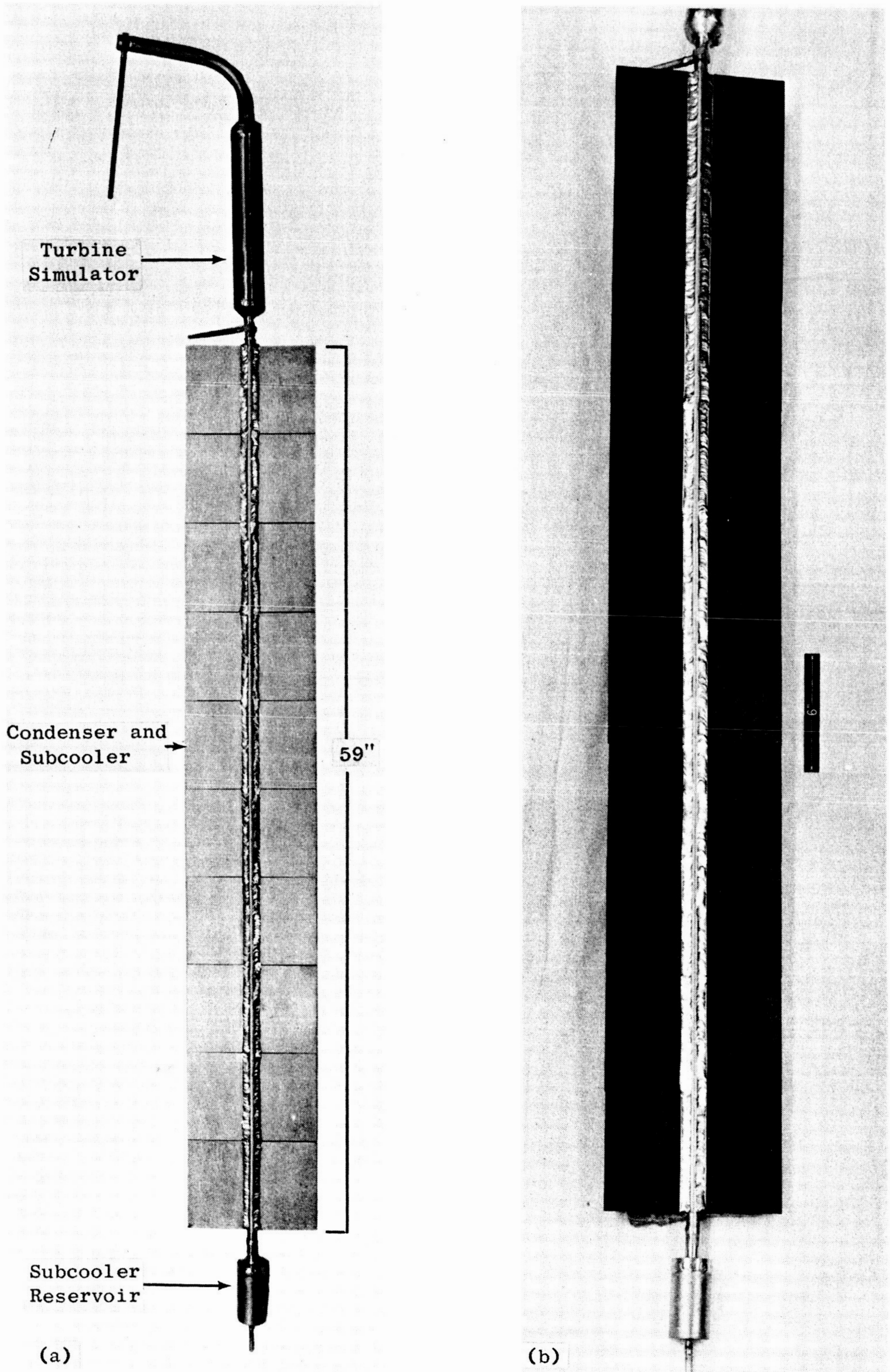


Figure 17. Turbine Simulator, Condenser/Subcooler and Subcooler Reservoir of the Prototype Corrosion Loop Before (a) and Following (b) Application of the Iron Titanate Coating.

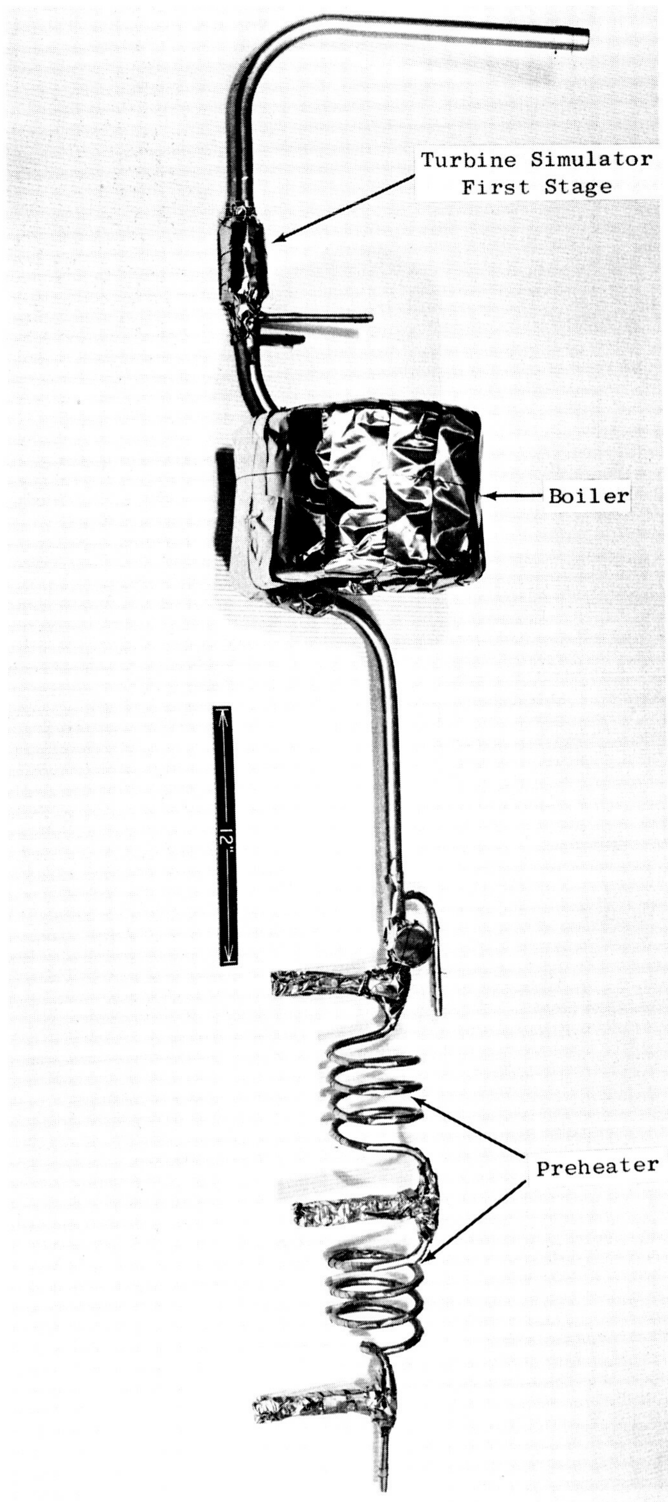


Figure 18. Preheater/Boiler/First Stage Turbine Simulator Assembly Wrapped with Cb-1Zr Alloy Foil Prior to Annealing. (Orig. C65031235)

one and one-half hours. During this time, furnace pressure increased to 8 x 10^{-5} torr at 1200°F, then decreased to 2.5 x 10^{-5} torr at 2200°F. During the one-hour annealing run at 2200°F, the pressure decreased to 8 x 10^{-6} torr. Approximately three minutes from the end of the furnace run, a series of small pressure pulses indicated an intermittent electrical short between a component and the furnace elements. The run was immediately terminated. After overnight cooling, the subassemblies were removed from the furnace and the point of electrical contact was found at the end of the process tube on the sodium heater coil. No harm was done since this tube had excess length which was removed before final assembly.

During the postweld annealing operation, the area between the nine stage turbine simulator and the condenser bent slightly causing a misalignment of these components. This condition was corrected by manual straightening in the welding chamber using a tungsten inert arc torch to heat the bent region.

10. Condenser Fin Coating Application

The use of a high emittance coating of iron titanate (Fe_2TiO_5) was suggested by the NASA Contract Manager to increase the heat rejection capability of the 0.25-inch thick x 4-inch wide x 60-inch long Cb-1Zr condenser fin. A NASA sponsored study currently underway at Pratt & Whitney Aircraft (11) has indicated that this coating has attractive properties. Test specimens were coated by R. Emanuelson of Pratt & Whitney Aircraft and evaluated by General Electric for thermal cycling characteristics under test conditions quite similar to those of the Prototype Loop.

Most of the results of the coating evaluation test were reported in the previous progress report (12). No changes in the temperature profile as a result of thermal cycling or test duration were noted. The surface appearance of the coated specimen following the evaluation test is shown in Figure 19(a) and (b). No significant alteration in the before and after test appearance was detected by examination with a steromicroscope at 30 diameters. Cross-sections of the coating and Cb-1Zr substrate before and after test are shown in Figure 19(c) and (d). Some evidence of cracking and interface separation is apparent in the tested fin in the high temperature (1500°F) region. Areas of the fin which were at lower temperatures (1070°-1280°F) had the same metallographic appearance as the before test specimen. Since no changes were detected in the temperature profile as a function of test time, the deterioration observed had a negligible effect on the heat rejection characteristics of the coating.

After successful conclusion of these tests, a stainless steel mockup of the condenser was fabricated and shipped to Pratt & Whitney Aircraft for use in establishing techniques for coating of the condenser fins.

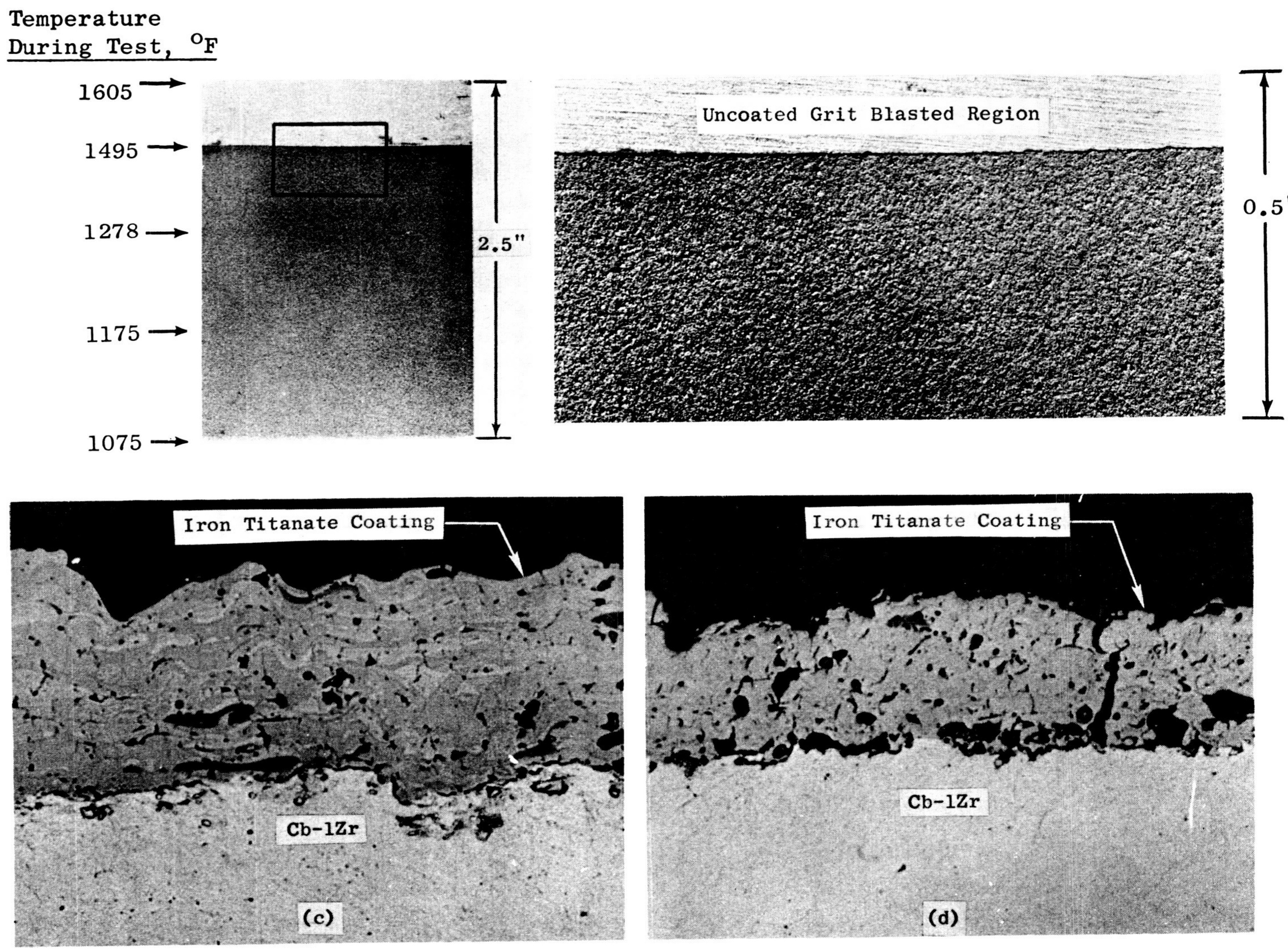


Figure 19. Cb-1Zr Alloy Fin Specimen Coated with Iron Titanate. The Appearance of the Surface of the Coating at Low Magnification After the 1,000-Hour Evaluation Test is Shown in (a) and (b). Metallographic Cross Sections of the Coating Before Test (c) and After Test (d) are also Included.

Prior to application of the iron titanate coating to the Cb-1Zr fin, the surface of the Cb-1Zr fin was grit blasted with 50-micron alumina. Each fin side was grit blasted just prior to application of the coating. Previous coating tests indicated that poor adherence of the coating to the metal surface has resulted when there is a considerable time delay (in excess of a few hours) between the grit blasting and coating operations. During grit blasting and coating, aluminum tape was used to mask off other regions of the condenser. The coating was applied by means of a plasma-arc device (Plasmatron) utilizing argon to form the plasma. Conditioning the surface by going over it completely with the argon plasma only (no iron titanate feed into the plasma) has also been found to promote adherence and this approach was used. Each pass gave an iron titanate deposit 1/2-inch to 3/4-inch wide, and after each pass of the gun back and forth over the length of the fin, the gun was manually indexed about 1/4 inch. Each pass laid down a deposit about 1/2 mil in thickness. A total coating thickness of approximately 3 mils was obtained. The temperature of the back side of the condenser fin was monitored during application of the coating and did not exceed 300°F. The coated condenser subassembly is shown in Figure 17(b).

C. Alkali Metal Purification and Transfer Systems for the Prototype Corrosion Loop

The approach which has been used in the preparation of the sodium and potassium for the Prototype Loop has been to procure the highest quality alkali metals, subject them to purification techniques designed to further reduce the impurity concentrations, transfer them to the loops using equipment and techniques designed to reduce the possibility of recontamination to a minimum, and finally to re-analyze the alkali metal after it has been flushed through the loop to insure its purity.

1. General Description of Purification and Transfer Equipment

Figure 20 shows a simplified schematic of both the sodium and potassium purification and transfer systems, and Figure 21 shows their spatial arrangement in relation to the Prototype Loop and test chamber. Referring to Figure 20, the processing steps proceed from left to right. The steps are as follows:

Sodium	Potassium
a. Filtration	a. Filtration
b. Hot Trapping	b. Hot Trapping

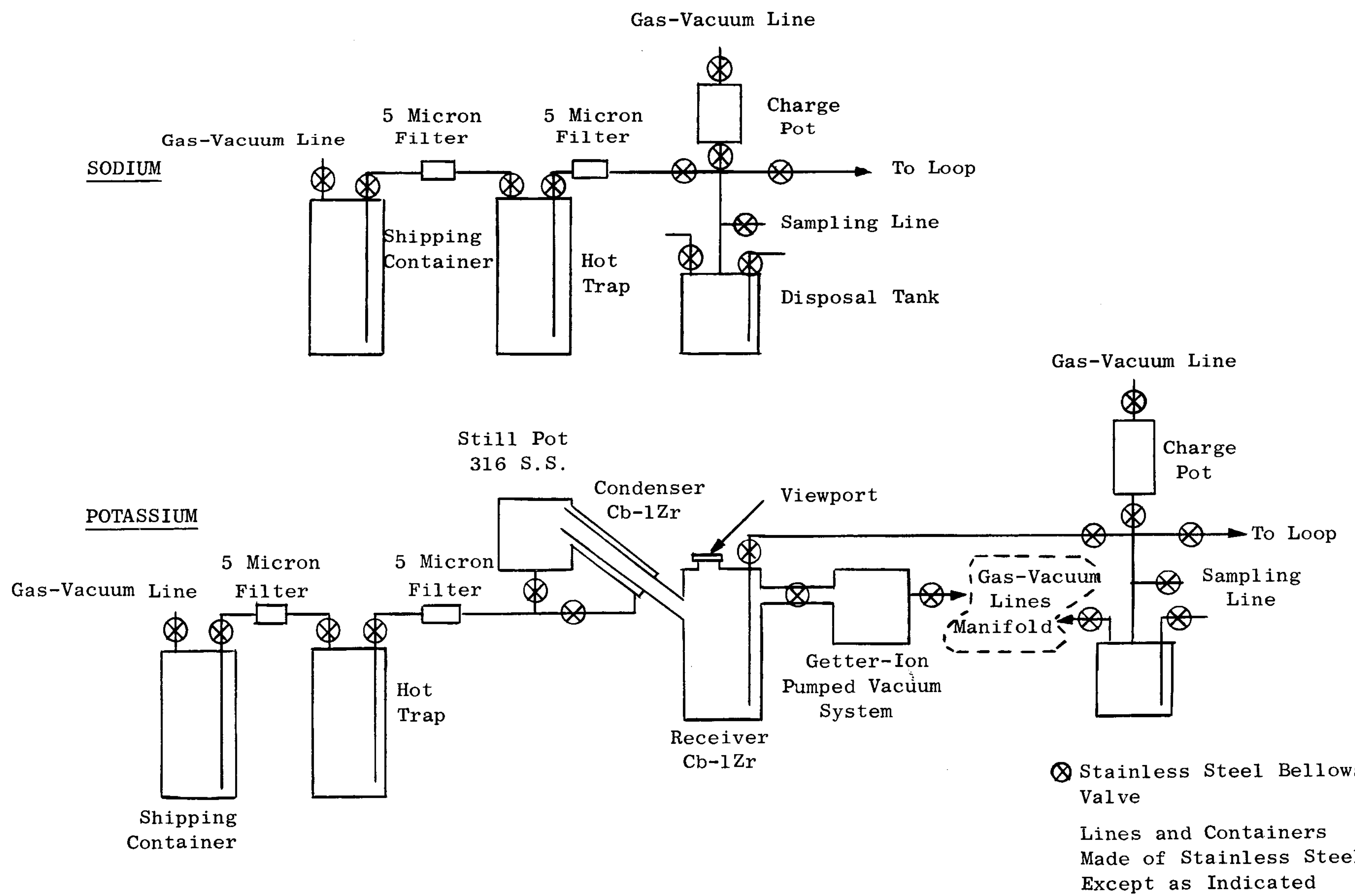


Figure 20. Simplified Schematic of Sodium and Potassium Purification and Transfer System for the Prototype Corrosion Loop.

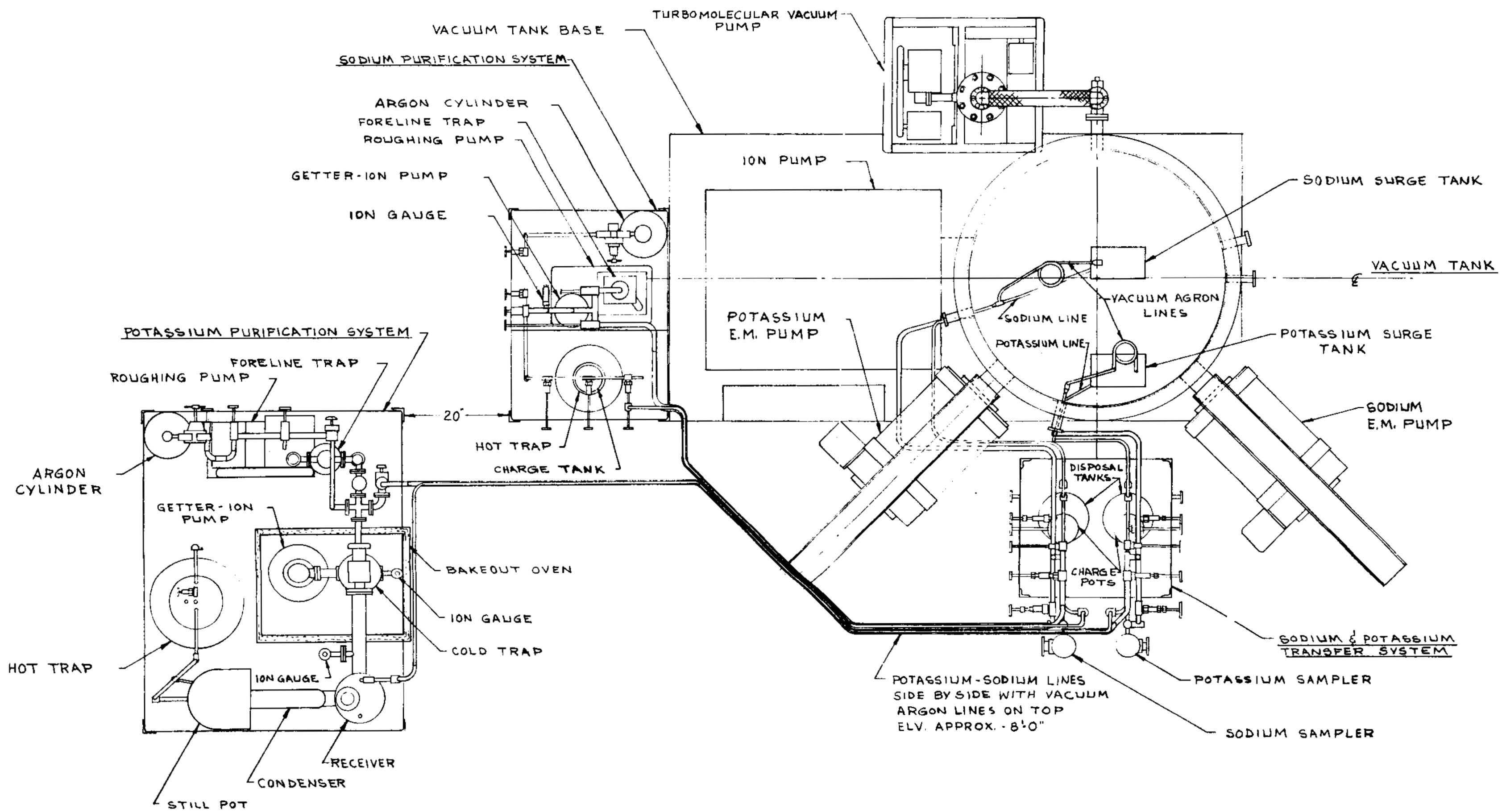


Figure 21. Plan View Showing Spatial Arrangement and Interconnecting Lines Between Purification Systems, Transfer Systems and Loop.

Sodium		Potassium	
c.	Filtration	c.	Filtration
d.	Transfer to Charge Pot	d.	Distillation
e.	Transfer to Loop	e.	Filtration
		f.	Transfer to Charge Pot

The main purpose of the Prototype Loop is to study corrosive and erosive interactions between potassium and the secondary circuit components which are fabricated of specially selected alloys; consequently, the potassium will be distilled as well as hot trapped in order to reduce the residual metallic impurities to a minimum. Further more, the potassium will contact stainless steel only at temperature less than 300°F after it has been distilled. During distillation, the condensing vapor and distillate will contact Cb-1Zr alloy only. The distillation will be performed under vacuum with conditions such that only surface evaporation takes place, i.e., actual boiling is avoided. In this way, recontamination of the distillate by the carry over of liquid drops is avoided.

Filtration will be performed during each transfer operation except for the final transfer from the charge pots to the loops. These filters have a nominal pore size of 5 microns and should prevent the transfer of particulate matter which may be left, inadvertantly, in any purification component or which may be produced during the purification steps.

Every effort is made to produce and maintain an immaculately clean system and all components are helium leak checked after fabrication and after assembly. The total leak rate of the entire system must be less than 5×10^{-10} std cc of air per second.

2. Sodium Purification System

The sodium purification system used for the Prototype Loop is the same one as used previously for Loop II. This system is shown before final closure in Figure 22 and after final assembly in the electrically heated oven in Figure 23. The control panel is shown in Figure 24. The hot trap consists of a Type 316 stainless steel body, containing a titanium liner and a zirconium getter, and is fitted with a vacuum-inert gas line, thermocouple well, and dip leg. The ratio of the surface area of the 0.012-inch thick zirconium foil to the mass of sodium in the hot trap is 1 $in^2/5$ gm. The dip leg is connected to a charge pot through a positive return valve. All valves are of the bellows sealed type. Angle type valves are used wherever the possibility exists that sodium must be cleaned out of them, and they are oriented so that the bellows are on the hot trap side of the seat. This arrangement permits them to be

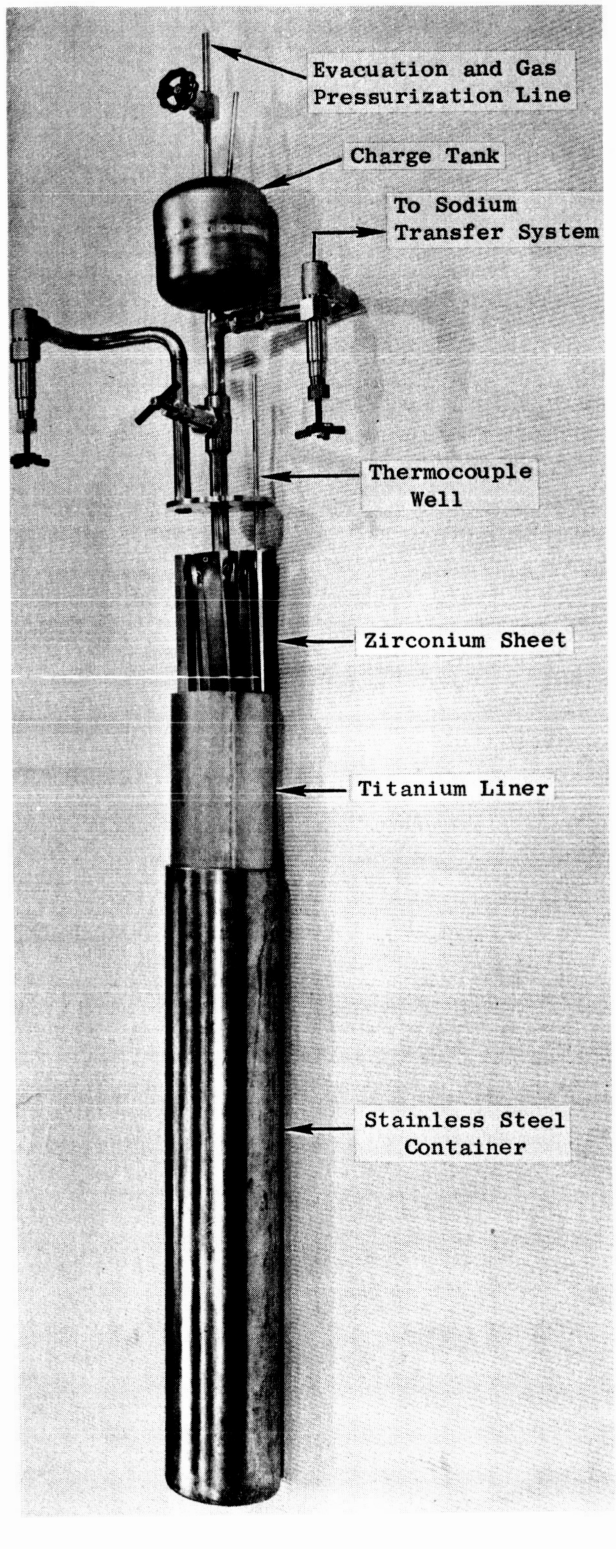


Figure 22. Sodium Hot Trap and Charge Tank. (Orig. C64041327)

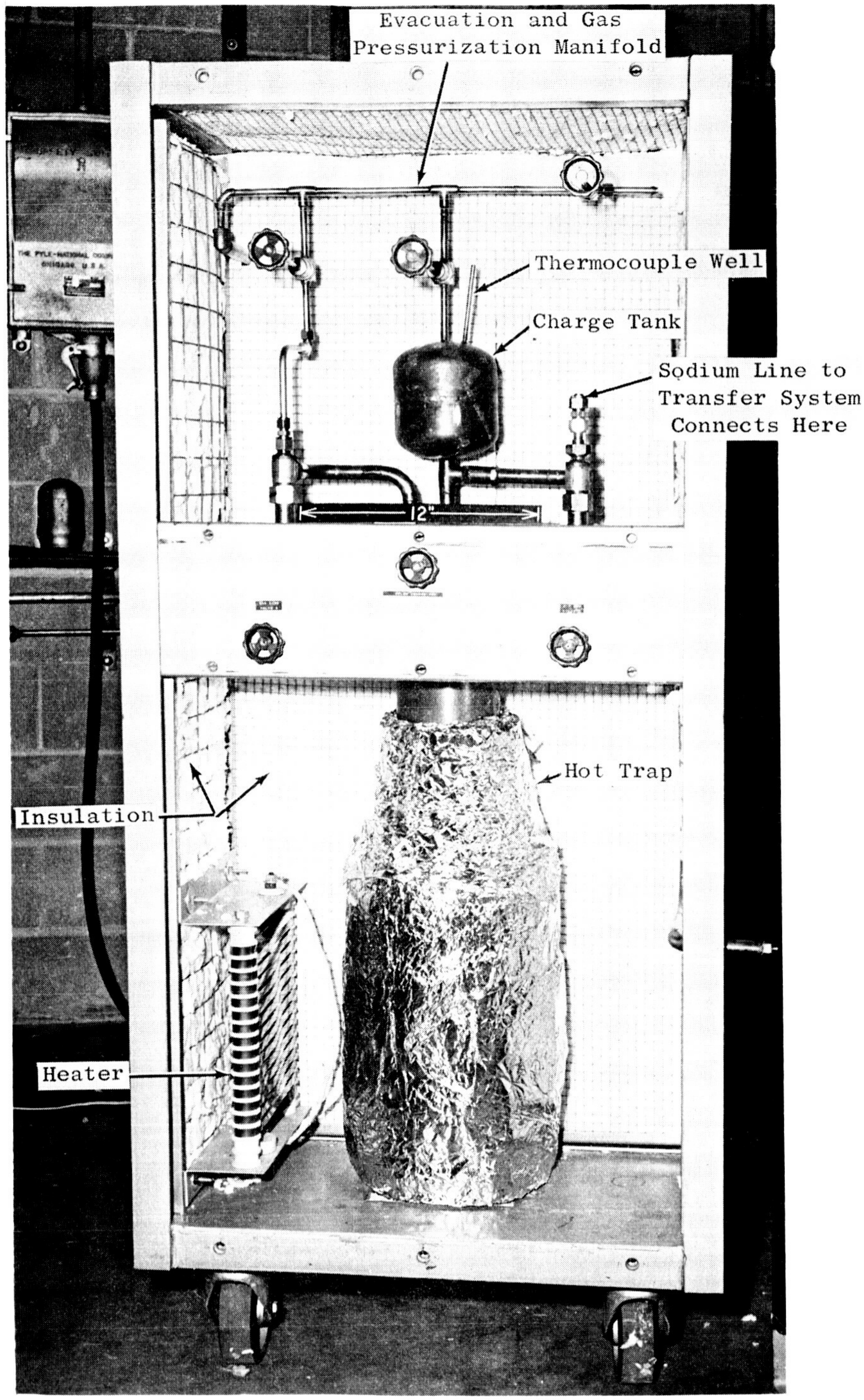


Figure 23. Sodium Purification System Showing Hot Trap and Charge Tank After Installation in Oven. Cover Panels Removed to Take Picture. (Orig. C65041402)

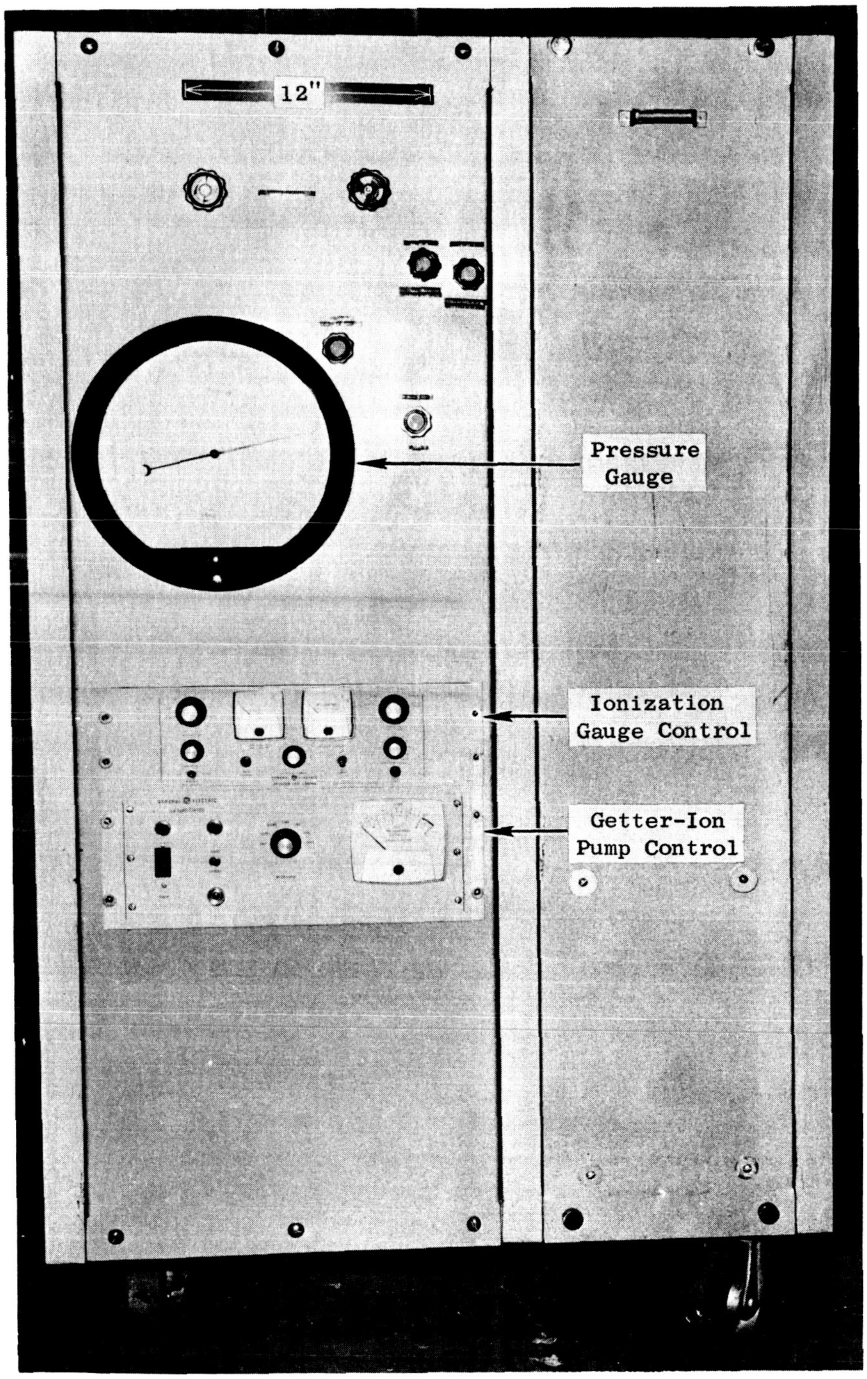


Figure 24. Control Panel of Sodium Purification System for the Prototype Corrosion Loop. (Orig. C65041401)

completely and easily cleaned back to the seat whenever it is necessary. All components of the hot trap excepting the titanium liner and zirconium getter are made of austenitic stainless steel. The oven is used to maintain the components at a temperature above the melting point of sodium during the transfer.

The vacuum system which is used to evacuate the system consists of a 25 liter per second, triode type, getter-ion pump backed up by a molecular sieve sorption trap and a 140 liter per minute mechanical vacuum pump. Matheson ultra high purity argon is used as a cover gas.

3. Sodium Purification

The sodium for the Prototype Loop was hot trapped in the system just described at 1300° to 1400°F for 48 hours. The purified product was analyzed for oxygen using the amalgamation method and for metallic impurities using emission spectrography. The results are shown in Table VI. It will be noted that all elements analyzed for, excepting oxygen, calcium, and silicon, are below the detection limits; and that the concentrations of those elements which were detected were extremely low.

4. Potassium Purification System

The potassium purification system consists of two major components: the hot trap and the still. The hot trap is shown in Figures 25 and 26 before and after assembly. It consists of a Type 316 stainless steel body containing a titanium liner and a zirconium getter and is fitted with a fill line, gas line, dip leg, thermocouple well, and a liquid level measuring tube. The ratio of the surface area of the 0.012-inch thick zirconium foil to the mass of potassium in the hot trap is 1 in^2/5 gm. The same type of valving is used as was used for the sodium hot trap. The dip leg is fitted with a stainless steel filter having a nominal pore size of 5 microns.

Liquid metal volumes will be measured by the pressure-volume technique and checked by thermal gradient measurements along the thermocouple wells.

The potassium still is shown unassembled and assembled in Figures 27 and 28, respectively. It consists of a stainless steel still pot, a coaxial tube type, air cooled condenser, the inner tube of which is Cb-1Zr, and a Cb-1Zr receiver. The selection of the coaxial type condenser was based on a desire to prevent the distillate from contacting any metal except Cb-1Zr at high temperature.

Heat rejection in the condenser is accomplished by conduction across the liquid potassium in the annular space between the two tubes. The potassium will be forced into this space from the hot trap prior to distillation

TABLE VI. CHEMICAL ANALYTICAL RESULTS ON SODIUM FOR THE PROTOTYPE CORROSION LOOP FOLLOWING THE HOT TRAP PURIFICATION TREATMENT

Element	Chemical Analysis ppm
O_2	4, 9
Ag	<1
Al	<1
Ca	10
Cb	<1
Co	<1
Cr	<1
Cu	<1
Fe	<1
Mg	<1
Mn	<1
Mo	<1
Na	--
Ni	<1
Pb	<1
Si	1
Sn	<5
Ti	<1
V	--
Zr	<5

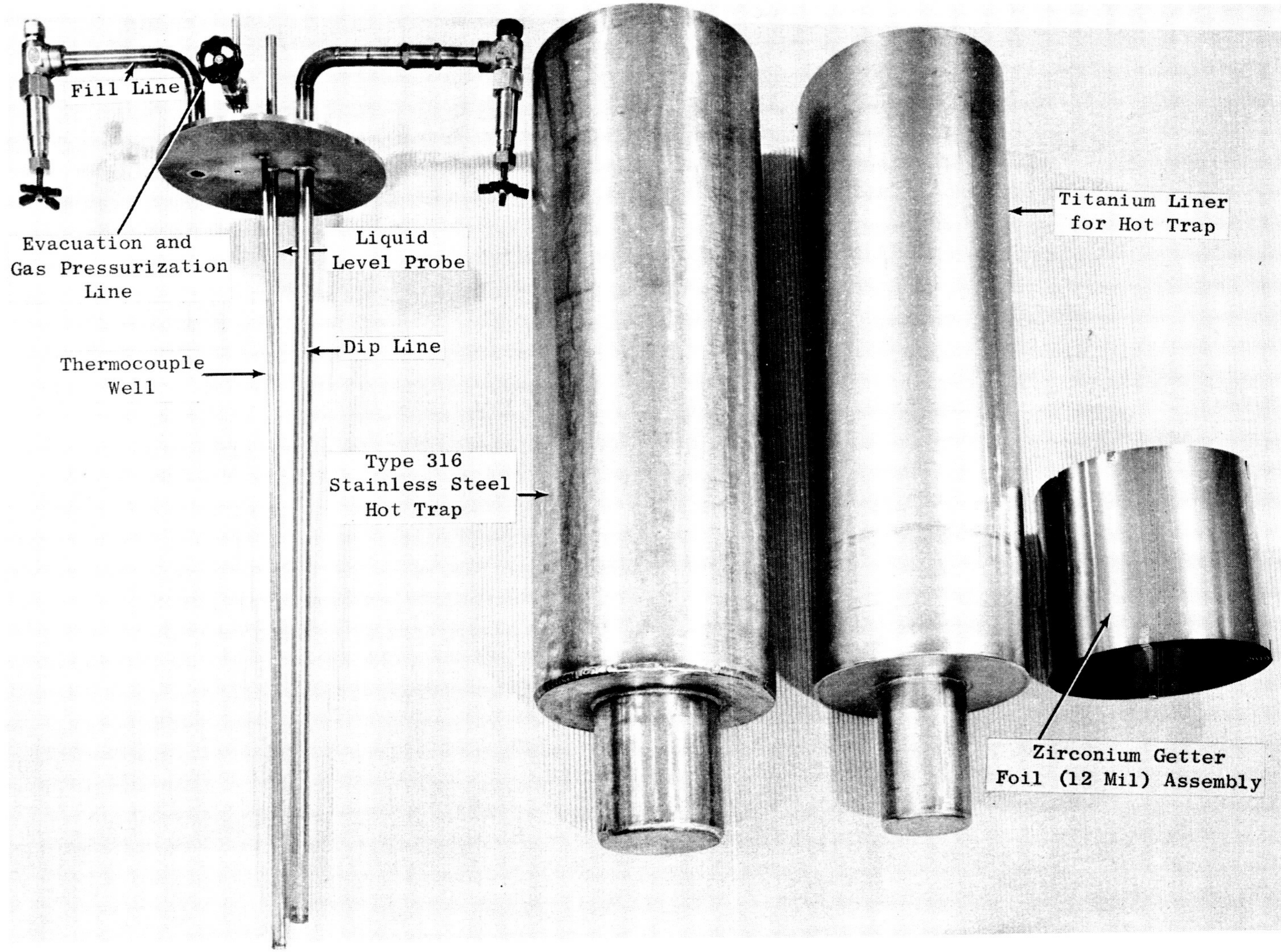


Figure 25. Potassium Hot Trap for the Prototype Corrosion Loop Prior to Assembly. Type 316 Stainless Steel Container (10" Schedule 40 Pipe), Titanium Liner (0.040" Thick) and Zirconium Foil (0.012" Thick) Are Shown.

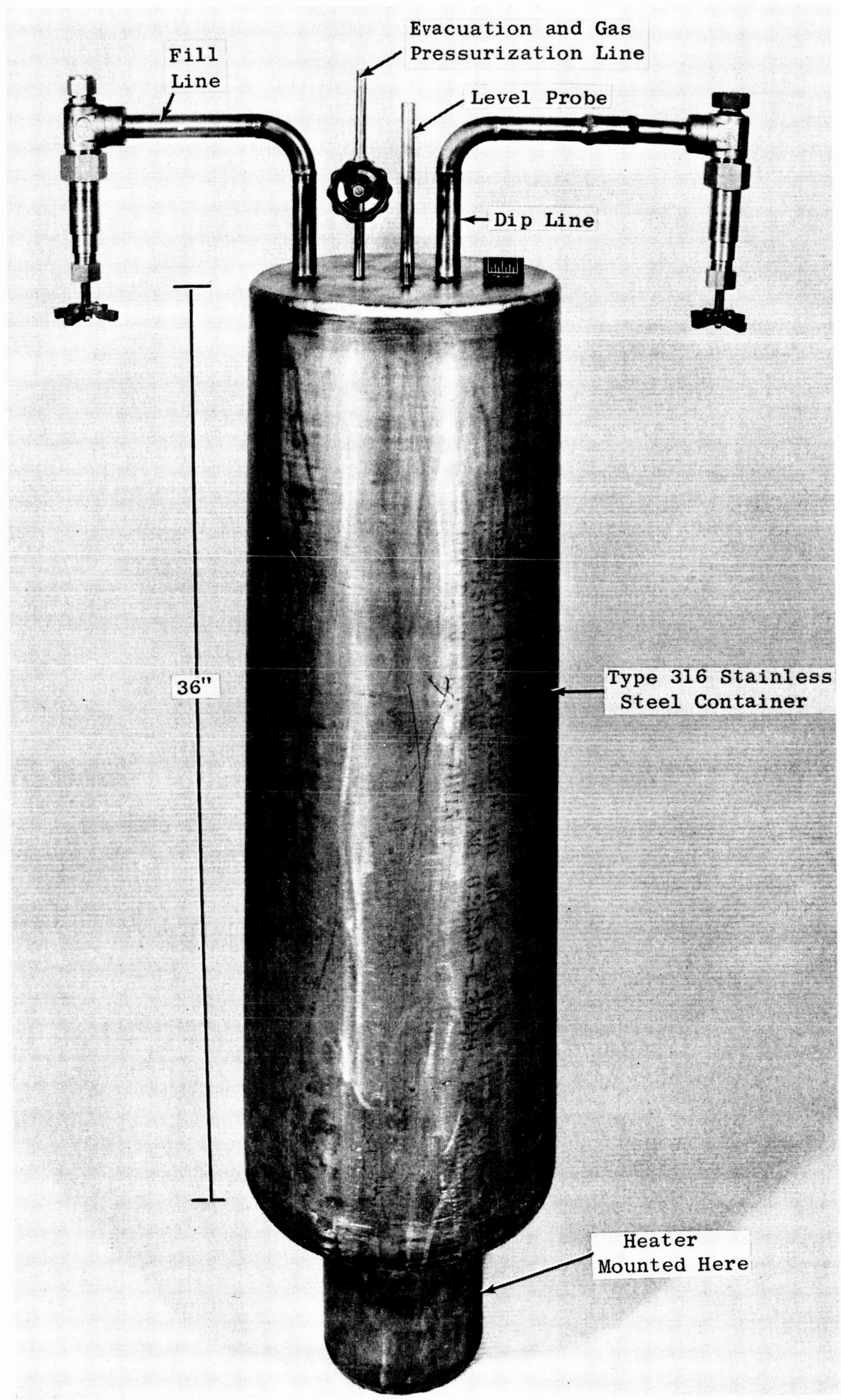


Figure 26. Hot Trap for Purifying Potassium for the Prototype Corrosion Loop. Hot Trap has a Capacity of 50 Pounds of Potassium. (Orig. C65021030)

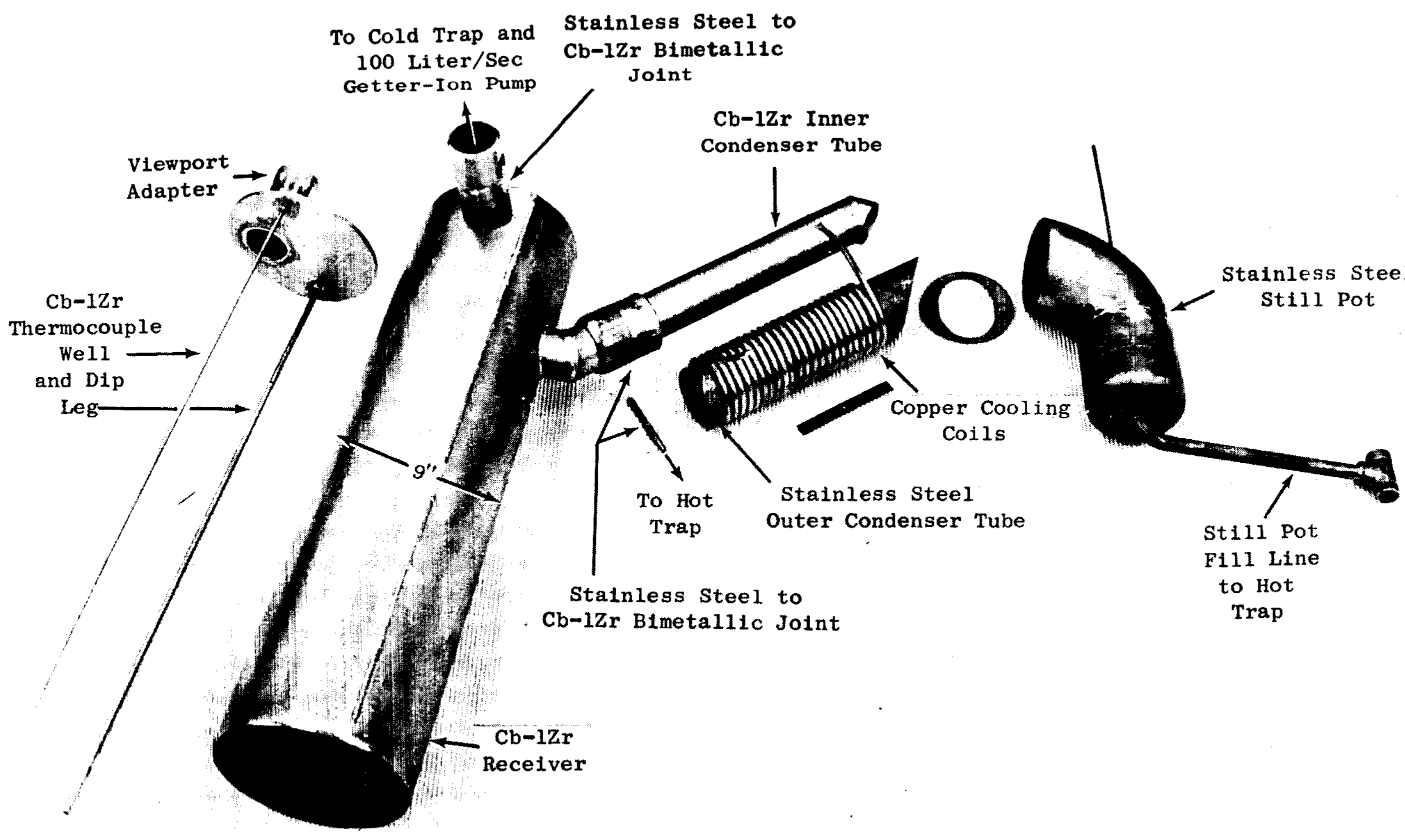


Figure 27. Potassium Distillation Unit for Purifying Potassium for the Prototype Corrosion Loop Test. Components of System Shown Prior to Assembly. (C65062186)

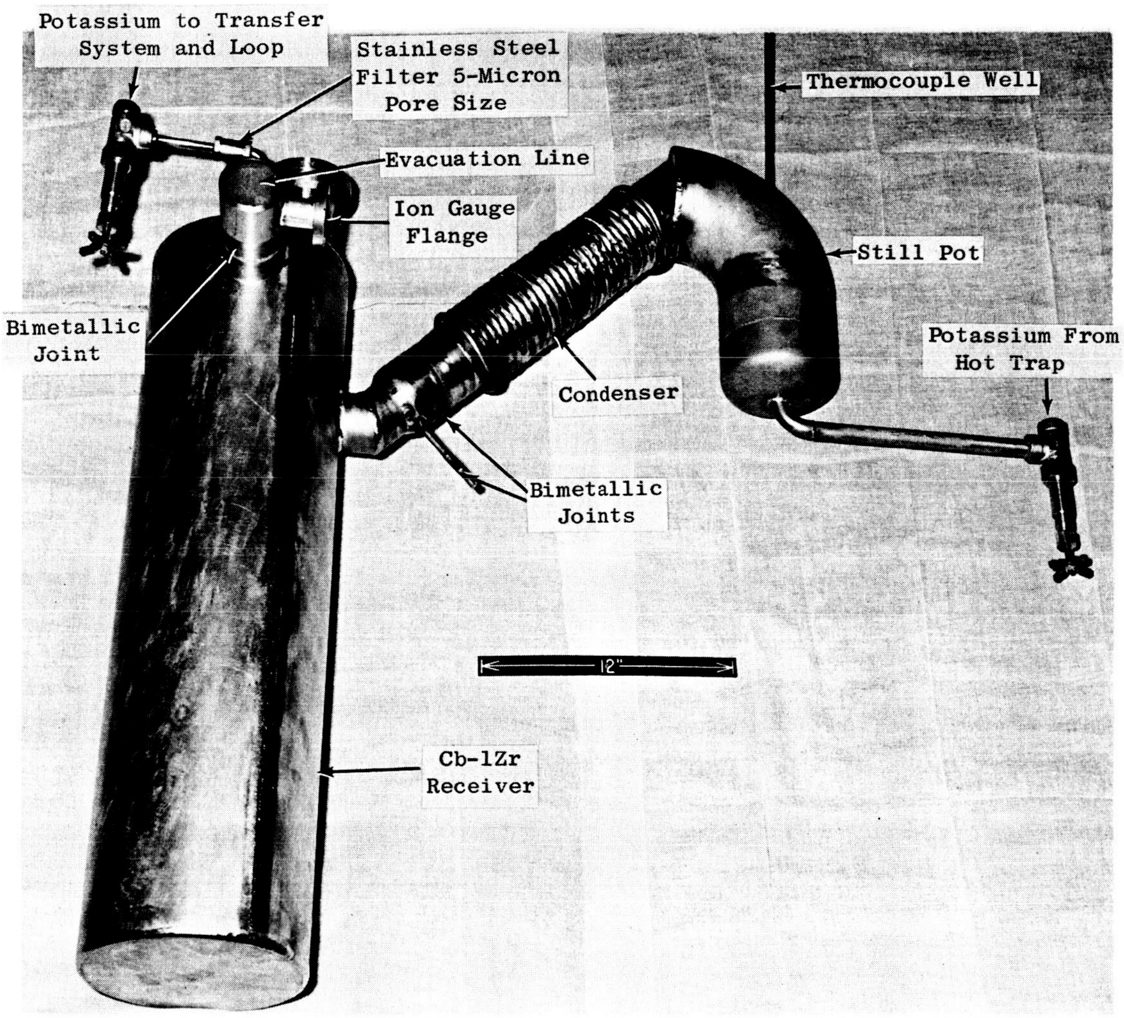


Figure 28. Potassium Distillation Unit After Assembly. (Orig. C65041404)

by means of the small tube shown at the exit end of the condenser. The details of the construction of the entry end of the coaxial condenser are shown in Figure 29.

The still pot is fitted with a fill line and a thermocouple well, and the receiver is provided with a vacuum line, sight port, thermocouple well, and dip leg. A spillway or trough extends from the exit of the condenser to the bottom of the receiver to minimize splash. Also, an optical baffle is provided to prevent line of sight evaporation from the exit of the condenser to the vacuum line. Stainless steel to Cb-1Zr brazed joints are used to make the transition between the two alloys.

The vacuum system for the potassium purification system consists of a 100 liter per second, triode type, getter-ion pump backed up by a cryogenic sorption pump and a 140 liter per minute mechanical pump. A liquid nitrogen cooled optical trap is provided between the high vacuum valve to the still receiver and the getter-ion pump to prevent diffusion of potassium vapor to the pump.

This high vacuum system and the associated vacuum-argon manifold were assembled, interconnected and mounted on the dolly as shown in Figure 30. The system was pumped down to 5×10^{-6} torr and then helium leak checked. No leaks were detected using a helium mass spectrometer having a sensitivity of 5×10^{-11} std cc of air per second. The hot trap and still will also be mounted on this dolly.

A variety of valves has been used in the vacuum system and the vacuum-argon manifold. Their selection was based on conductance and vacuum requirements, maximum bakeout temperature, and whether bakeout was to be performed with the valve open or closed. All components are made of austenitic stainless steel except for the Viton O-rings which are used in some of the valves, and the copper gaskets used in flanges. Interconnections are made by welding or using ConFlat* flanges.

5. Sodium and Potassium Transfer Systems

The sodium and potassium transfer systems are mirror images of one another and are mounted in the same oven enclosure as shown in Figure 31. All components and lines are made of austenitic stainless steel. The charge pots are sized to deliver the required amount of liquid metal to the loop surge tanks.

----

* Varian Associates, Palo Alto, California.

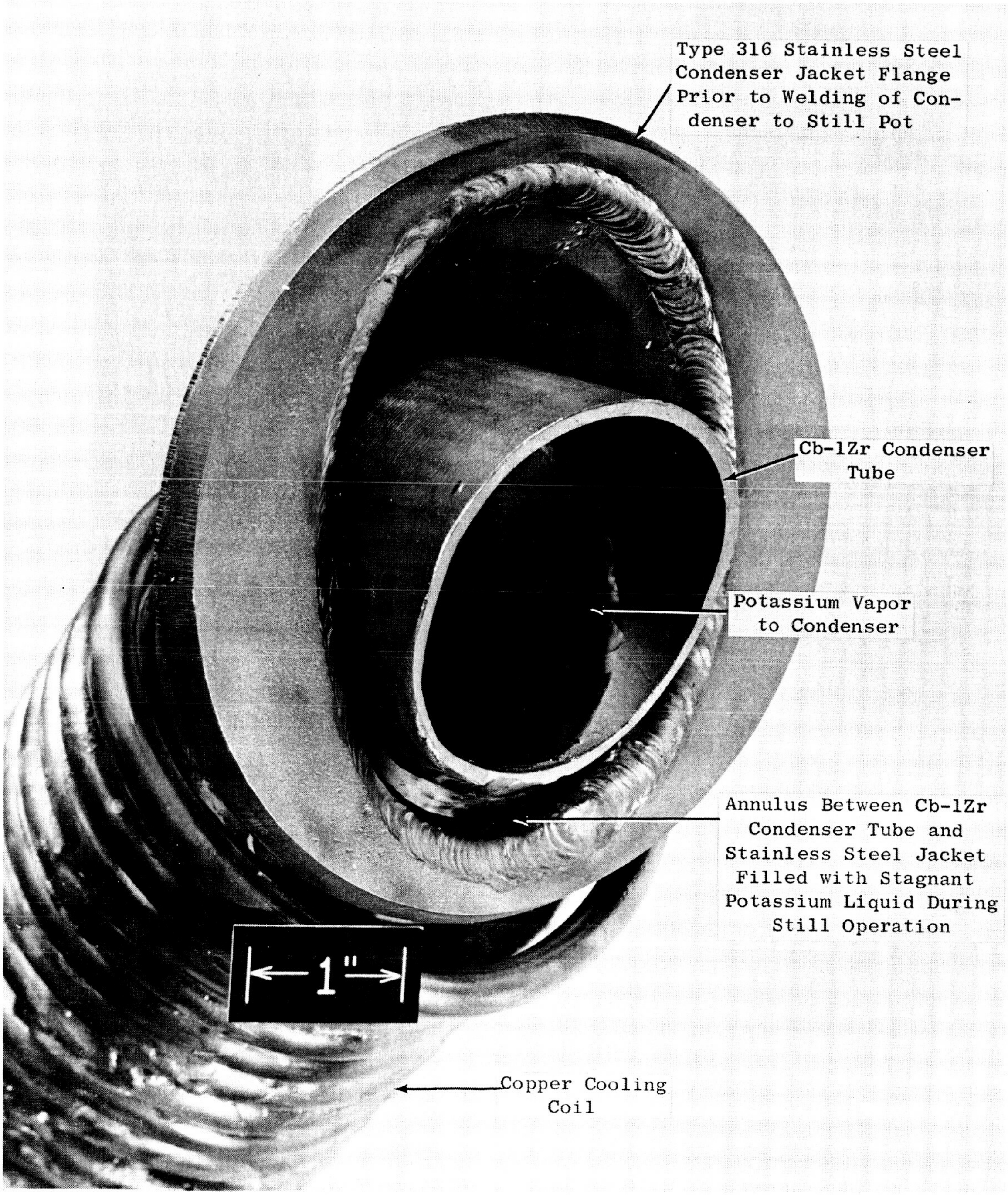


Figure 29. High Vacuum Potassium Still, Partially Assembled, Showing Annular Arrangement of Cb-1Zr Inner Tube and Type 316 Stainless Steel Outer Tube of Condenser. Potassium in the Annular Space Provides Heat Transfer for Condensing. (Orig. C65040929)

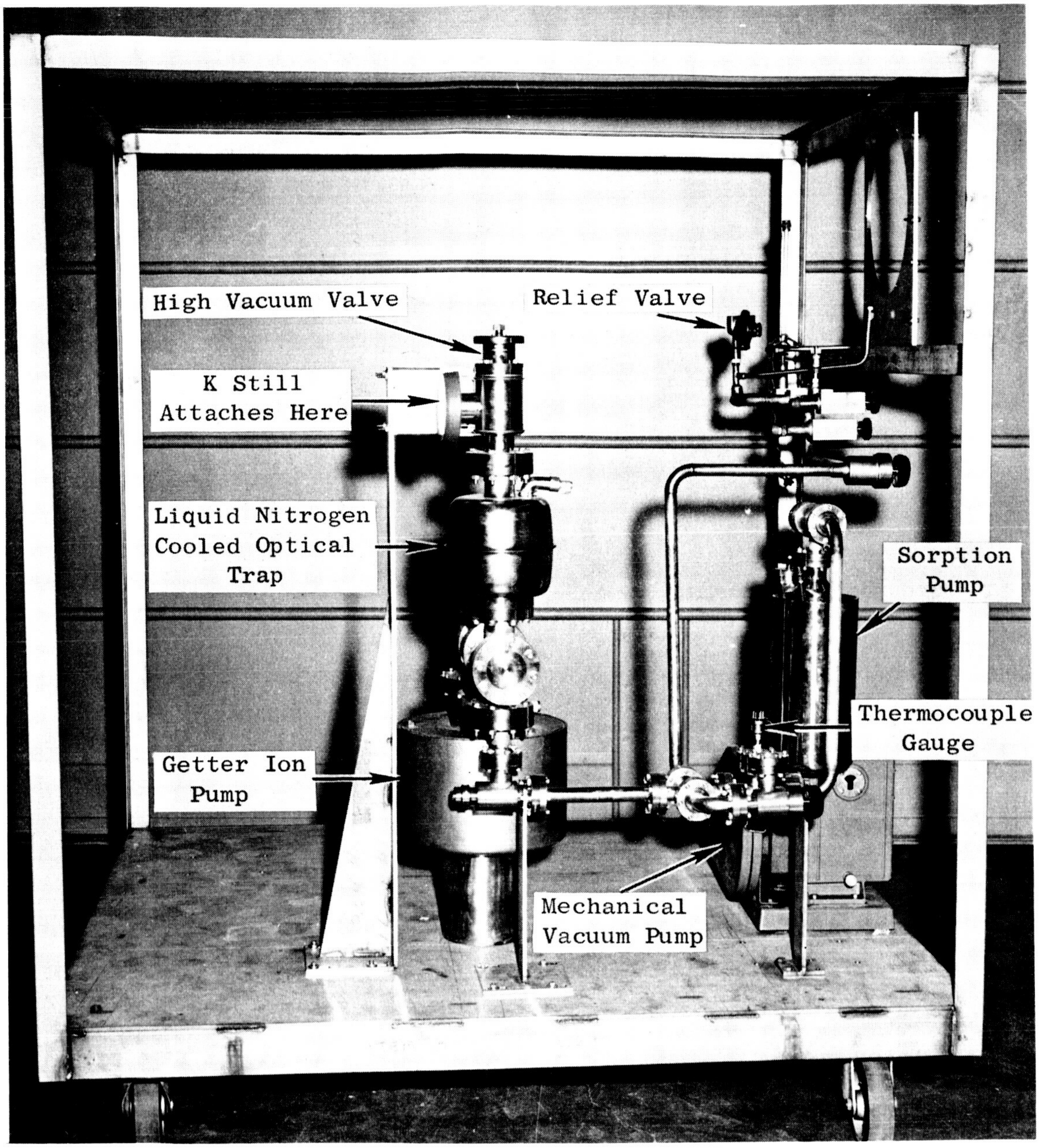


Figure 30. Assembled High Vacuum System for the Potassium Purification System. (Orig. C65041403)

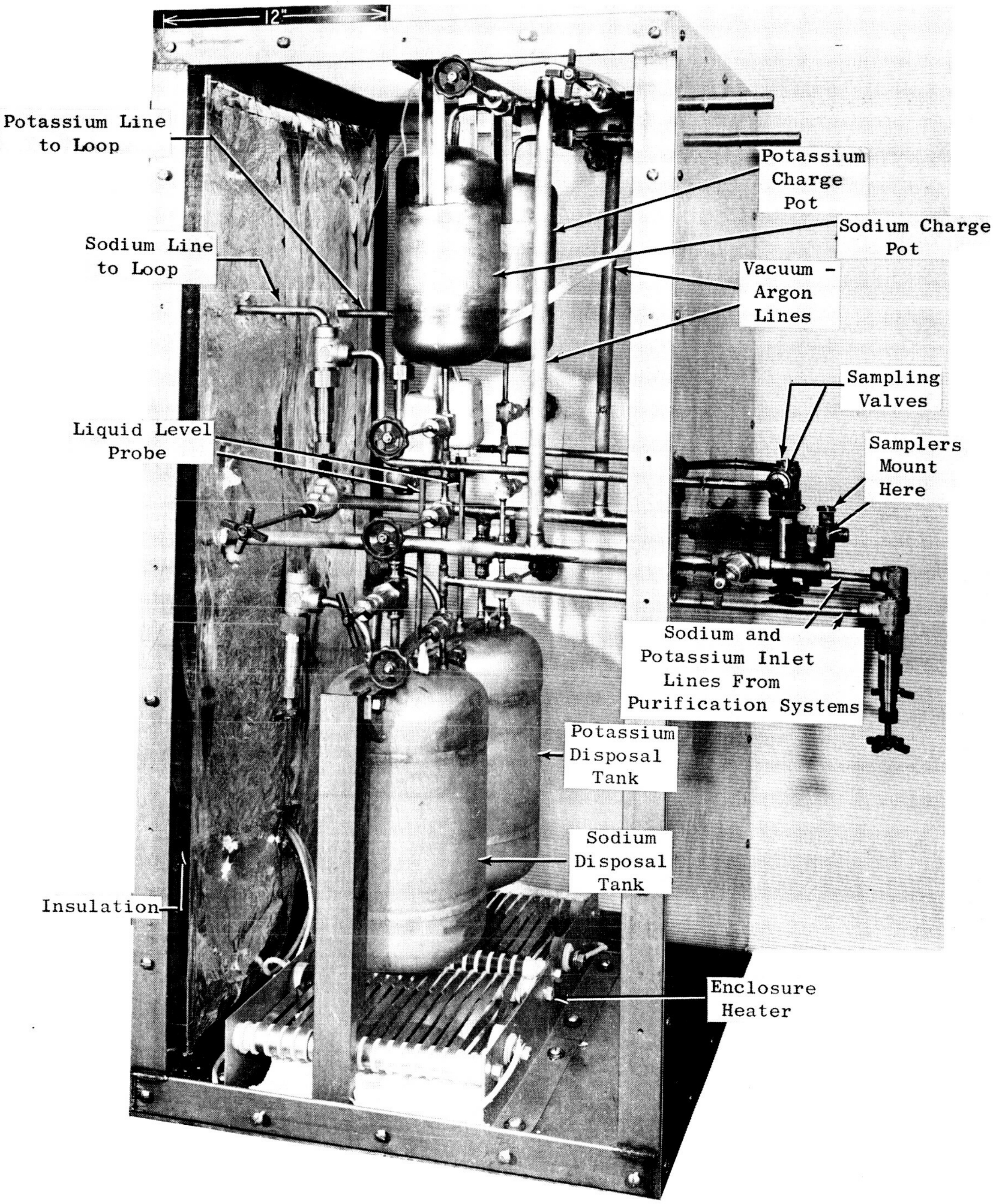


Figure 31. Sodium and Potassium Transfer Systems for the Prototype Corrosion Loop After Installation in Oven Enclosure. Cover Panels Removed to Take Picture. (Orig. C65040926)

6. Alkali Metal Sampling Devices

An assembled alkali metal sampler is shown in Figure 32. It consists of a stainless steel body which is actually an overflow reservoir, capped by a sight port and fitted with a fill line and vacuum-argon line. The fill line penetrates the bottom of the reservoir such that the top of the fill line is at about the same level as the entry port of the vacuum-argon line. In operation, a specimen tube is positioned between the Conoseal* tube union below the fill line valve and a similar union on the alkali metal sampling valve on, for example, the transfer system. The sampler is evacuated and sufficient alkali metal overflowed into the reservoir to flush the specimen tube and fill line. The sampler is then pressurized with argon, and the alkali metal is allowed to freeze. The specimen is pressurized during cooling to minimize the occurrence of voids. Both valves on the sampler may be completly disassembled to facilitate cleaning. The assembled sampler is evacuated and helium leak checked before taking additional samples.

7. Welding and Brazing Methods Used in Fabricating Components

The welding of stainless steel and Cb-1Zr was performed in accordance with specifications SPPS-41 and 3C, respectively, except that postweld heat treatments have not been performed since these components are intended for low temperature use only. Preparation of stainless steel to Cb-1Zr brazed joints has been in accordance with specification SPPS-9A. The cobalt-base braze alloy AMI-400** (General Electric Flight Propulsion Division Specification B50T56) was used to make the bimetallic joints. The titanium hot trap lines were welded under helium in the welding chamber under conditions comparable to those used for welding the Cb-1Zr components.

D. Prototype Corrosion Loop Components and Instrumentation

1. Partial Pressure Analyzer for Prototype Loop Chamber

The partial pressure analyzer for the Prototype Loop chamber has been adjusted for optimum performance and calibrated for hydrogen, helium, nitrogen and argon. The analyzer tube (GE Model 22PT120) has an exposed ion source and thus should yield partial pressure data corresponding closely to the actual pressures within the chamber due to the very high conductance between the chamber and mass spectrometer ion source. The analyzer tube is mounted on the chamber sump. The electronic control used to operate the new analyzer tube is a GE Model 22PC120, an improved version of the previously available

----

* Marmon Division, Aeroquip Corporation.

** Alloy Metals, Inc., Detroit, Michigan.

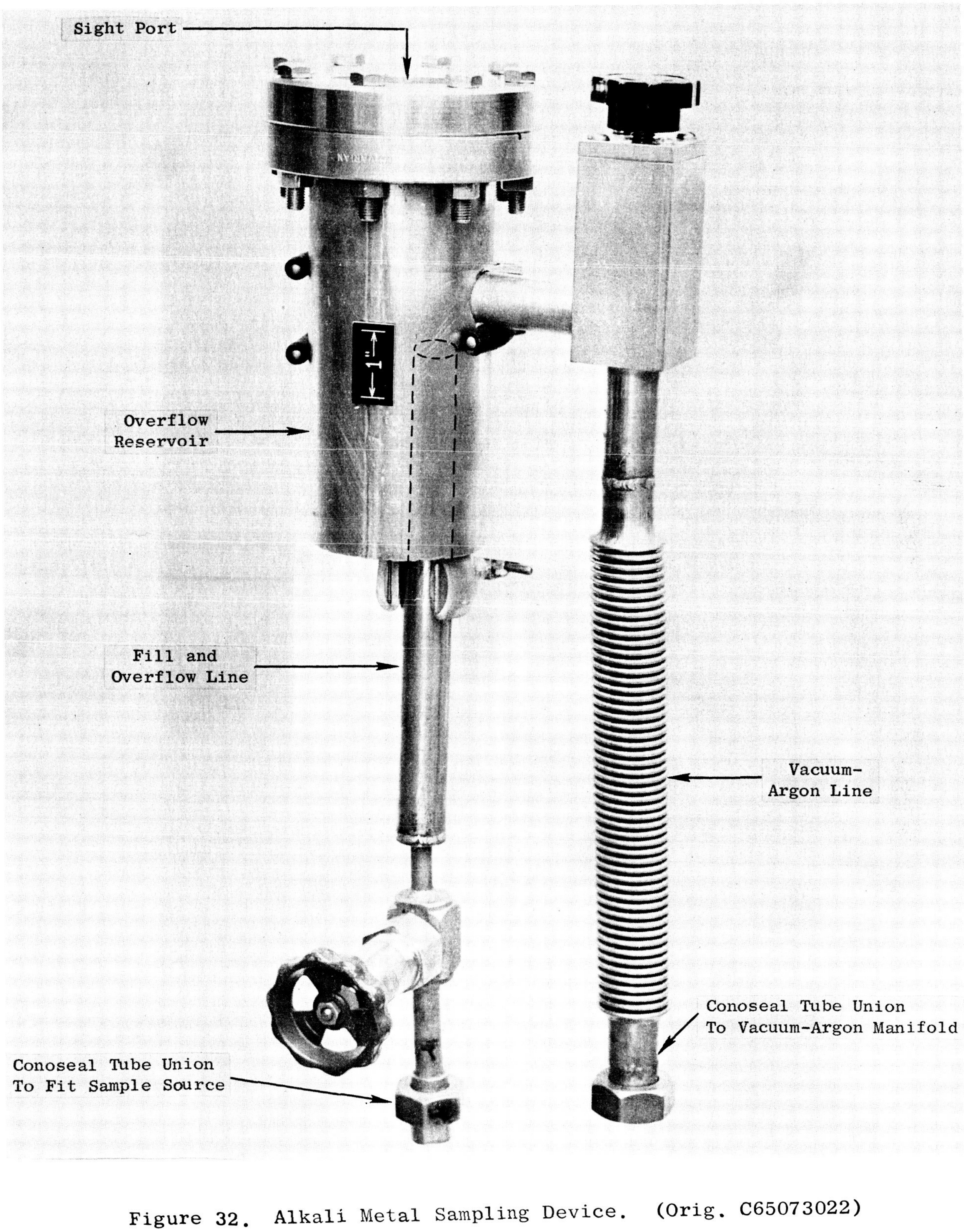


Figure 32. Alkali Metal Sampling Device. (Orig. C65073022)

unit. This new control has both logarithmic and linear readout, provisions for single peak monitoring, and improved stability in the accelerating voltage circuitry.

A baffle has been fabricated and located in the duct between the getter-ion pump and the chamber sump. This baffle is a circular piece of stainless steel sheet which, in the closed position, nearly blocks the opening to the pump. The baffle may be rotated around its diameter by a magnetically operated rotary feedthrough (Varian 954-5026) so that, in the open position, the total duct conductance is obtained.

This baffle may be partially or completely closed whenever it is desired to decrease the gas load to the getter-ion pump. The baffle is used on initial startup of the pump and during chamber bakeout when the gas load may be particularly high. The baffle was also closed during admission of gases used to calibrate the partial pressure analyzer to prevent excessive gas flow to the pump while maintaining relatively high chamber pressures. This was particularly useful for the helium and argon calibration since these inert gases are not effectively removed by the getter-ion pump.

The partial pressure analyzer calibration procedure has been described in a previous report (13). With the baffle closed, a pure gas was admitted to the chamber, a reading of the ion gauge was made, and a mass spectrum was obtained. This process was repeated at various pressures for each of the four calibrating gases. Appropriate corrections were applied when appreciable partial pressures of gases other than the calibrating gas were present. The average sensitivity of the partial pressure analyzer for each of the calibrating gases was then calculated. The sensitivity is here defined as the analyzer ion current divided by the pressure indicated by the ion gauge. The sensitivity for each of the four calibrating gases is plotted in Figure 33. Although a smooth curve is usually obtained with such a plot, in this particular case the helium sensitivity appears to be low. The sensitivity of gases for which no calibration has been made is obtained by extrapolating a smooth curve through the two points at higher masses (N_2 and A) as shown by the dashed line in Figure 33. This particular partial pressure analyzer has been found to be extremely sensitive, due principally to cesiation of the elctron multiplier by high temperature bakeout. The sensitivity to hydrogen is about a factor of 700 greater than that obtained for the analyzer used for the Loop II tests (13). Experience with partial pressure analyzers on the present program demonstrates the wide variation of sensitivity from one analyzer to another and also from one gas to another for a particular analyzer.

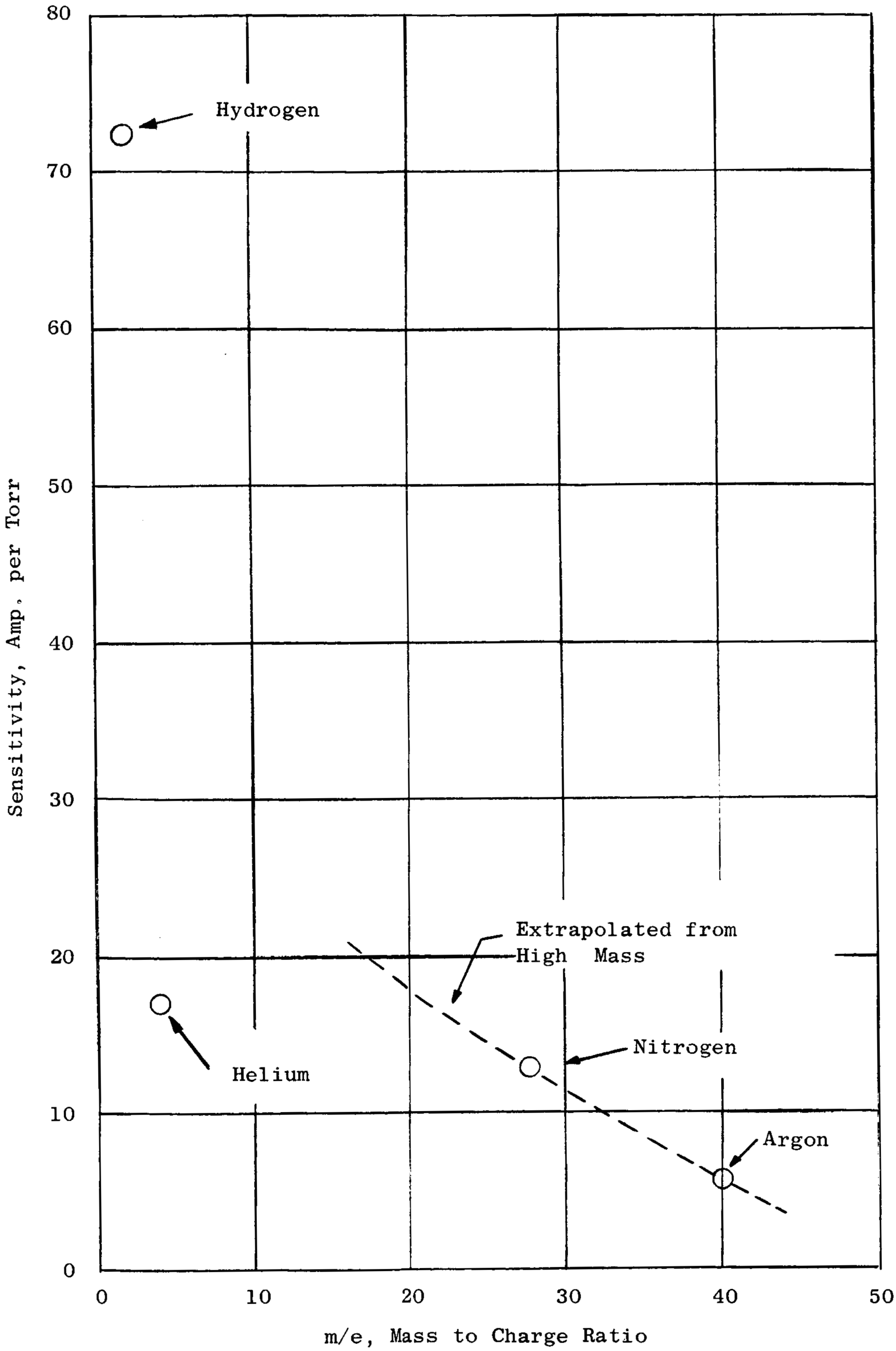


Figure 33. Sensitivity of Prototype Loop Partial Pressure Analyzer, Initial Calibration (3-3-65).

A summary of the data obtained from Figure 33 is given in Table VII along with values to be used to convert analyzer ion current to ion gauge partial pressure or to the true partial pressure.

In Table VIII are given partial pressures calculated from a typical mass spectrum showing the residual gases in the chamber with the baffle open. The peak at m/e = 20 is attributed to neon since the Ne_{22}^+ isotope was also observed. The calculated partial pressure of neon has been corrected for the contribution from A^{++}. Neon is not generally observed in residual gas spectra and its presence is a matter of some curiosity. It is interesting to note, however, that the ratio of the true neon pressure to that of helium is about the same as the ratio of the corresponding partial pressures in the atmosphere. The peak at m/e = 28 is attributed to N_2 since no peak at m/e = 12 was observed and the ratio of the 28 to 14 peak is the same as that obtained in the nitrogen calibration. Thus, the major residual gases are H_2, N_2 and Ne. The low H_2O and CO_2 indicate that the system has been well baked and is thus free of adsorbed gases. The high N_2 and relatively high O_2 indicates air leakage. The agreement between the ion gauge reading and the sums of both the ion gauge and true partial pressures is excellent.

2. Calibration of Refractory Alloy Thermocouple Wire in a Vacuum Furnace

Calibration of the thermocouple wire to be used in the loop test was conducted in a high vacuum environment with sample thermocouples made from the same spools of wire which will be used to instrument the Prototype Loop. The following is a detailed description of the apparatus used, the configuration of the calibration thermocouples, and a discussion of the test results, including data required to define a characteristic curve for the thermocouple wire.

The calibration was conducted in a vacuum furnace chamber constructed especially for the purpose. A sketch of this unit is shown in Figure 34. It consists of a 6-inch OD tube approximately 2 feet in length with appropriate high vacuum flanges to provide access to either end. A total of seven 1.5-inch ports were provided for power and thermocouple lead feedthroughs. An isothermal block, 2.6-inch OD and 15 inches long and made of Mo-TZM alloy, was mounted vertically in the center of the chamber. Various components of the calibration system are shown in Figure 35. Thermocouples to be calibrated were installed in a 0.5-inch hole drilled along the axis of the block approximately 7 inches deep from the lower end. A similar hole was provided from the top end of the block with a 0.25-inch web between the bottoms of the two holes. The upper hole was used for optical pyrometer readings of block temperature. Standard reference Pt/Pt-10%Rh thermocouples were installed in the bottom hole

TABLE VII. PROTOTYPE LOOP PARTIAL PRESSURE ANALYZER CALIBRATION I (3-3-65)

Parent Specie	m/e	Sensitivity Amp/Torr	K_i[1] Torr/Amp	Ion Gauge Sensitivity Relative to N_2	K_t[2] Torr/Amp
H_2	2	72.3	0.0138	0.42	0.033
He	4	16.9	0.059	0.19	0.031
CH_4	16	(21)[3]	0.048	1.07	0.045
H_2O	18	(19)[3]	0.053	0.89	0.060
Ne	20	(18)[3]	0.056	0.33	0.17
N_2 or CO	28	12.6	0.079	1.00	0.079
O_2	32	(10)[3]	0.10	0.84	0.12
A	40	5.74	0.17	1.56	0.11
CO_2	44	(3.7)[3]	0.27	1.37	0.20

[1] To obtain ion gauge partial pressure, multiply ion currenty by K_i.

[2] To obtain true partial pressure, multiply ion current by K_t.

[3] Extrapolated values from Figure 33.

TABLE VIII. COMPARISON OF ION GAUGE PARTIAL PRESSURES AND TRUE PARTIAL PRESSURES OF VARIOUS SPECIES IN THE PROTOTYPE LOOP CHAMBER[1]

Parent Specie	Ion Current Amp	Ion Gauge Partial Pressure[2] Torr	True Partial Pressure Torr
H_2	6.3×10^{-9}	0.87×10^{-10}	2.1×10^{-10}
He	0.16×10^{-9}	0.09×10^{-10}	0.50×10^{-10}
CH_4	0.1×10^{-9}	0.04×10^{-10}	0.045×10^{-10}
H_2O	0.03×10^{-9}	0.02×10^{-10}	0.018×10^{-10}
Ne	0.9×10^{-9}	0.50×10^{-10}	1.5×10^{-10}
N_2	2.2×10^{-9}	1.74×10^{-10}	1.7×10^{-10}
O_2	0.13×10^{-9}	0.13×10^{-10}	0.15×10^{-10}
A	0.32×10^{-9}	0.56×10^{-10}	0.35×10^{-10}
TOTAL OF PARTIAL PRESSURES:		3.95×10^{-10}	6.4×10^{-10}
ION GAUGE READING:		4.2×10^{-10}	4.2×10^{-10}

[1] Chamber cold and empty following bakeout.

[2] Ion Gauge Partial Pressure is the partial pressure uncorrected for the sensitivity of the ion gauge to the various gas species.

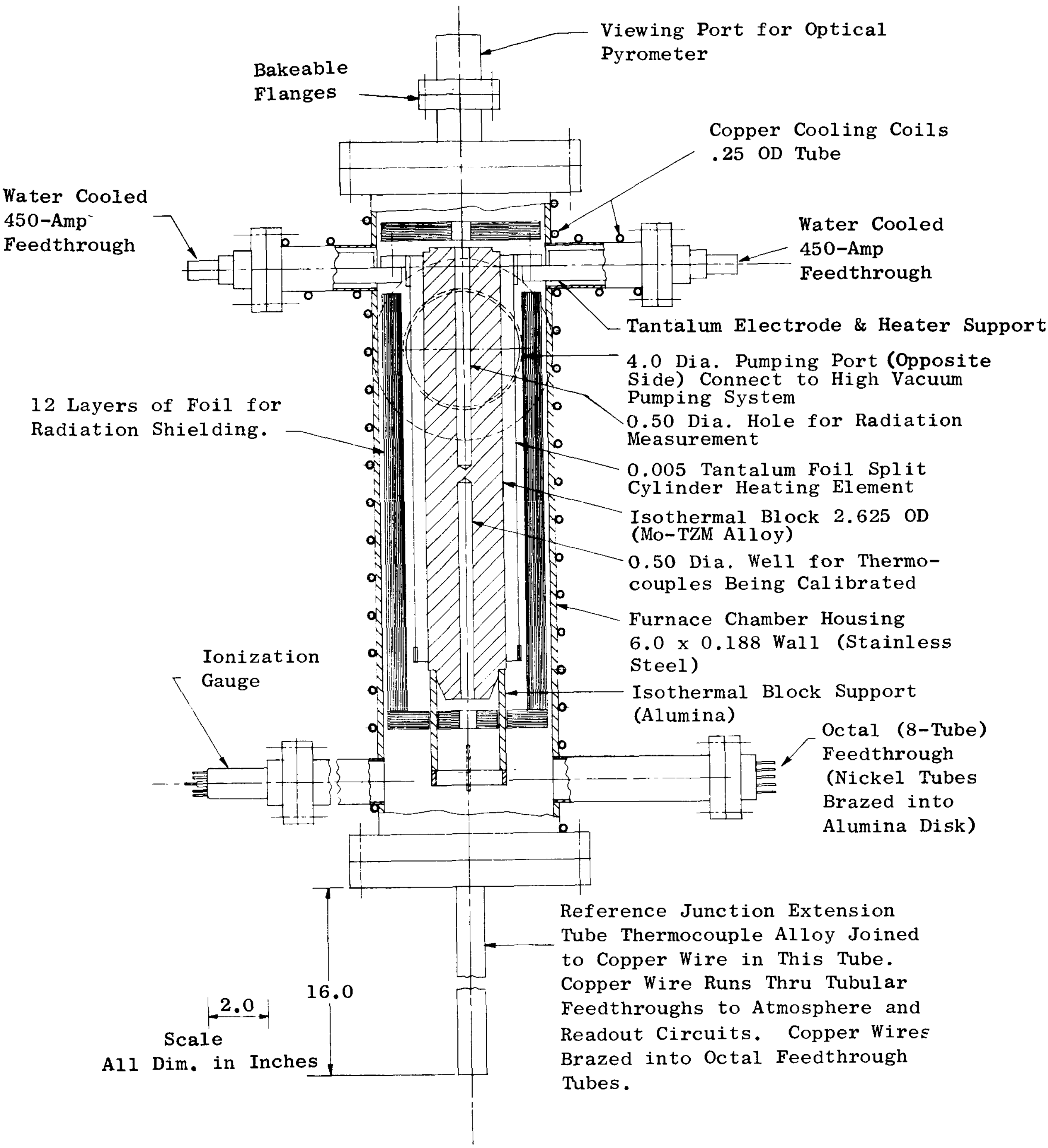


Figure 34. Vacuum Furnace for Calibrating W-3%Re/W-25%Re Thermocouple Wire.

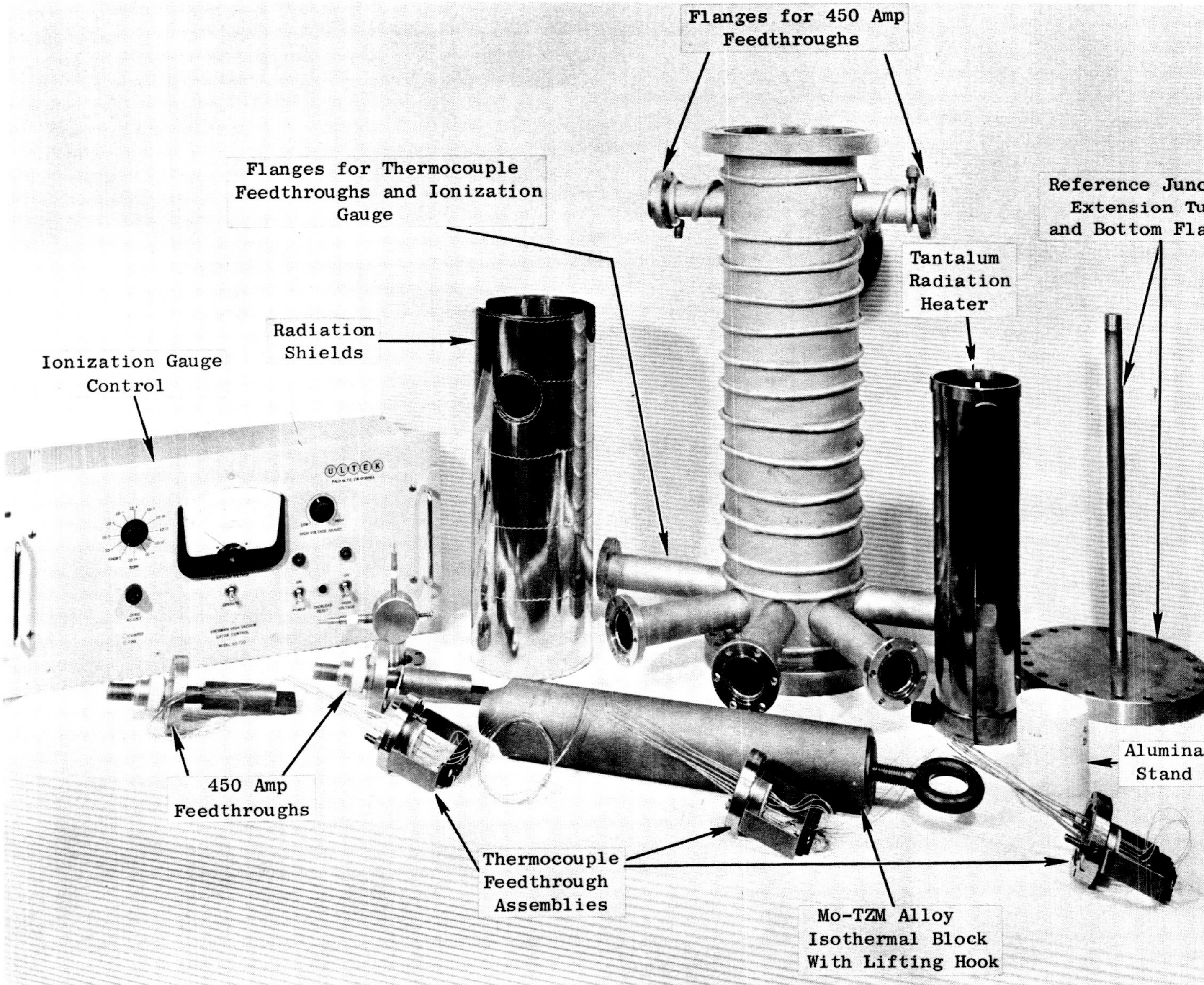


Figure 35. Components of Thermocouple Calibration Vacuum Furnace Before Assembly. (C65012206)

together with the W-3%Re/W-25%Re thermocouples to be calibrated. The optical pyrometer and the Pt/Pt-10%Rh thermocouples were used as independent means of measuring isothermal block temperatures.

The isothermal block was heated by means of a split cylindrical resistance element made from 0.005-inch thick tantalum foil. The element was supported on the 450-amp feedthroughs so that it was concentric with the isothermal block. Cylindrical radiation shields were provided to minimize the heat losses from the element to the walls of the chamber. A total of 12 shields were constructed of 0.005-inch thick foil separated by 0.020-inch tantalum wire. A view into the furnace chamber prior to installation of the isothermal block is shown in Figure 36.

The copper wire thermocouple leads were brought out through Varian nickel tube feedthroughs which consisted of eight 0.040-inch ID tubes brazed into an alumina disc which was brazed to a 2-3/4-inch OD high vacuum flange. The copper wires were brazed into the nickel tubes and connected to the thermocouple leads within the vacuum chamber in an extension tube welded to the bottom flange of the vacuum chamber. During calibration tests, the extension tube (1-inch OD x 16 inches long) containing the wire junctions was immersed in an ice bath to establish the cold junction reference.

A Varian Model UHV-14 nude ionization gauge was installed in one of the 1.5-inch OD tube extensions on the furnace chamber by means of a 2-3/4-inch OD flange. The furnace chamber was attached to the bell jar of a Varian vacuum system containing a 1000 liter/second ion pump as shown in Figure 37. Also shown in this picture are the switches and readout instrument used during the calibration. The readout instrument was a Honeywell Precision Indicator with an accurancy of $\pm$ 0.02% of any individual millivolt span from 1 to 70 with a 3-microvolt dead band.

An optical pyrometer was used to check the absolute block temperature. The 1.5-inch viewport window mounted on the upper chamber flange and the specific instrument lens had been calibrated and correction factors provided by calibration were used to compensate individual readings. Thermocouples to be calibrated were arranged as shown in Figure 38. Two Pt/Pt-10%Rh reference thermocouples made of 0.020-inch diameter wire were strung through a 4-hole 99% purity alumina insulator (0.187-inch OD). A piece of Cb-1Zr tubing (0.250-inch OD) was drilled out to make a tight fit and was slipped over the 4-hole insulator containing the Pt/Pt-10%Rh reference thermocouples. Samples of the W-3%Re and W-25%Re wire to be calibrated were strung through 2-hole 99% alumina insulators (0.062-inch OD) 30 inches long, and a 1-inch length of 2-hole 0.032-

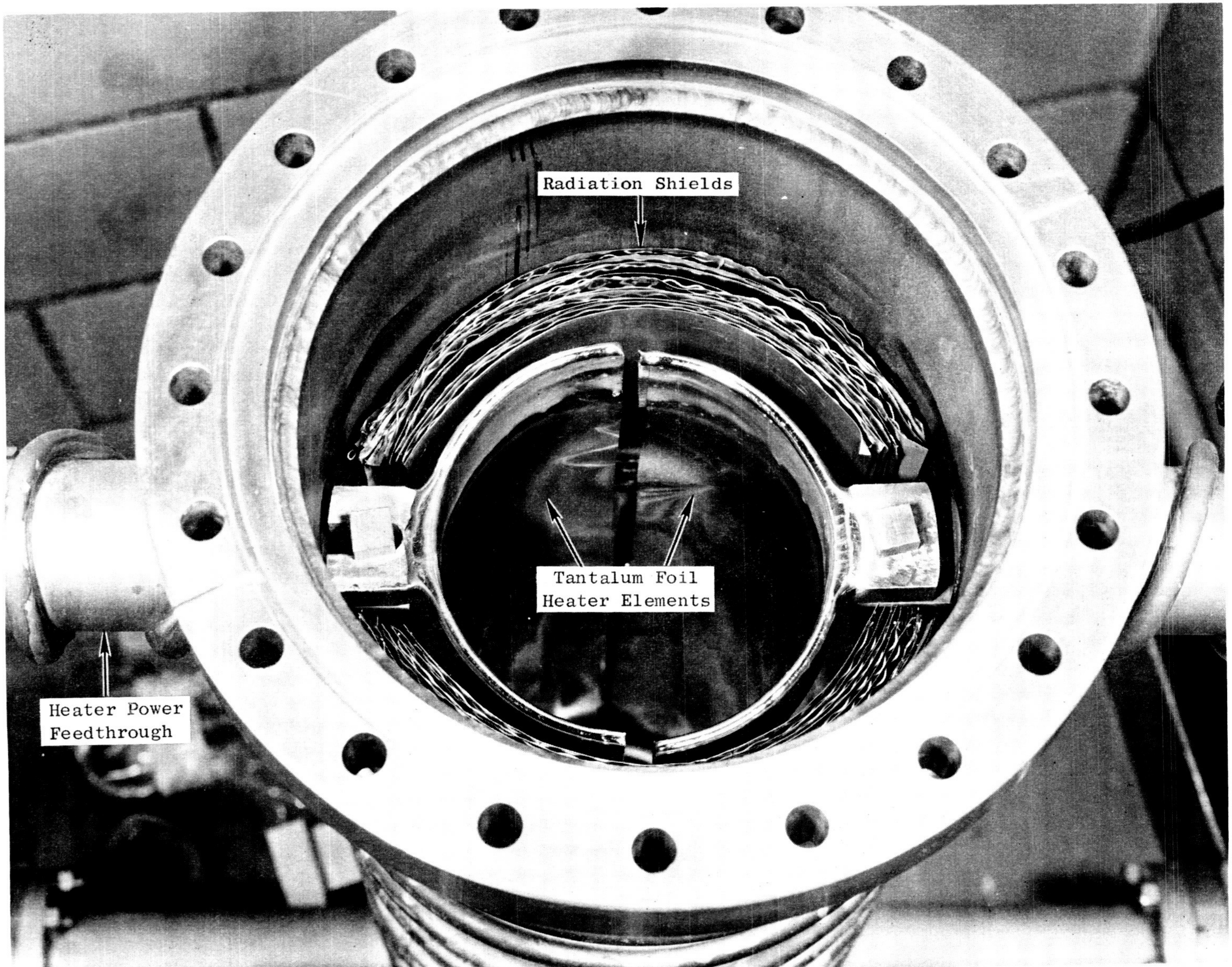


Figure 36. Inside of Thermocouple Calibration Furnace Prior to Insertion of Mo-TZM Alloy Isothermal Block. (Orig. C6502230)

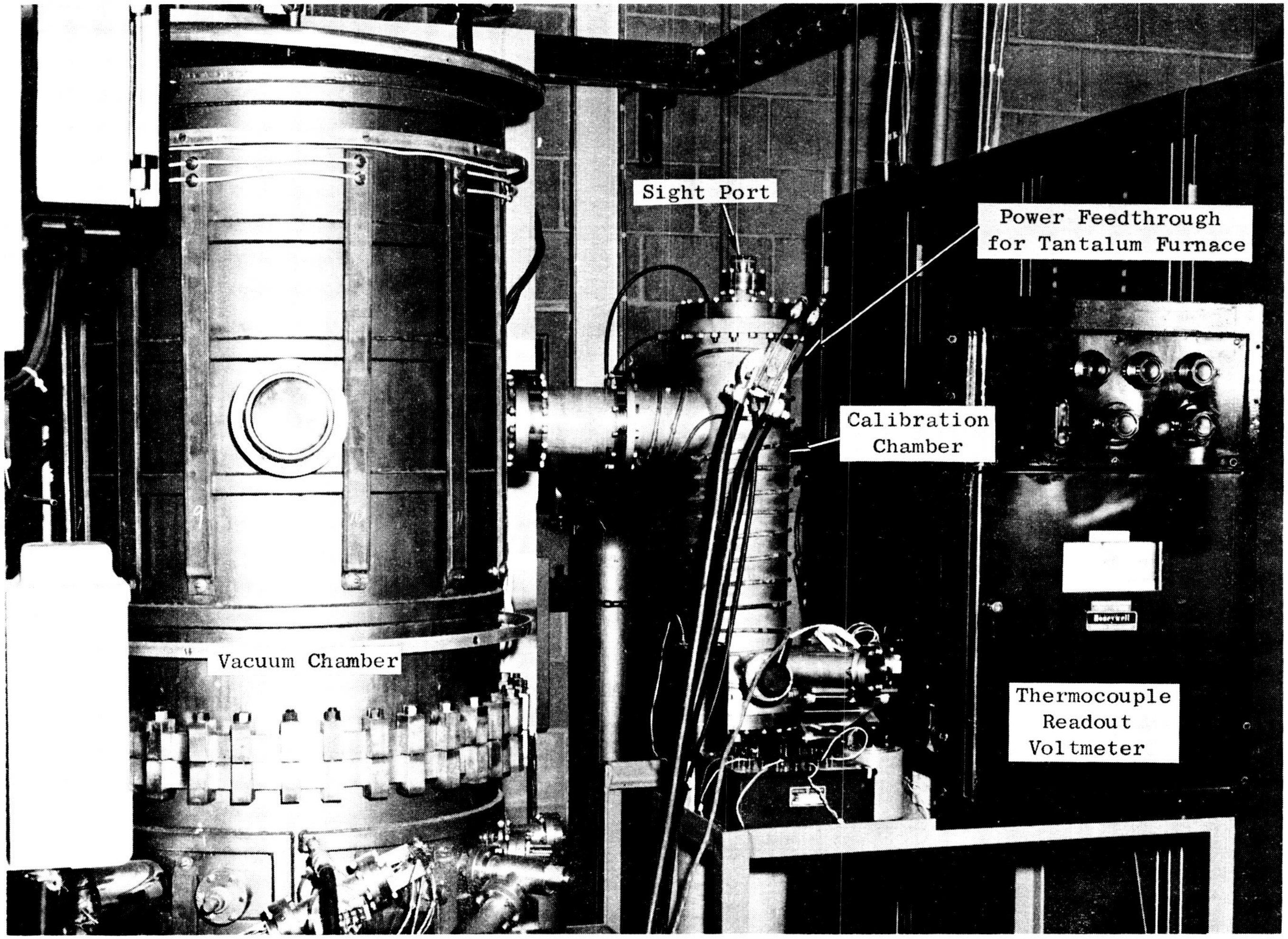


Figure 37. Thermocouple Calibration Furnace Attached to the 24-Inch Diameter Getter-Ion Pumped Vacuum Chamber. (Orig. C65040528)

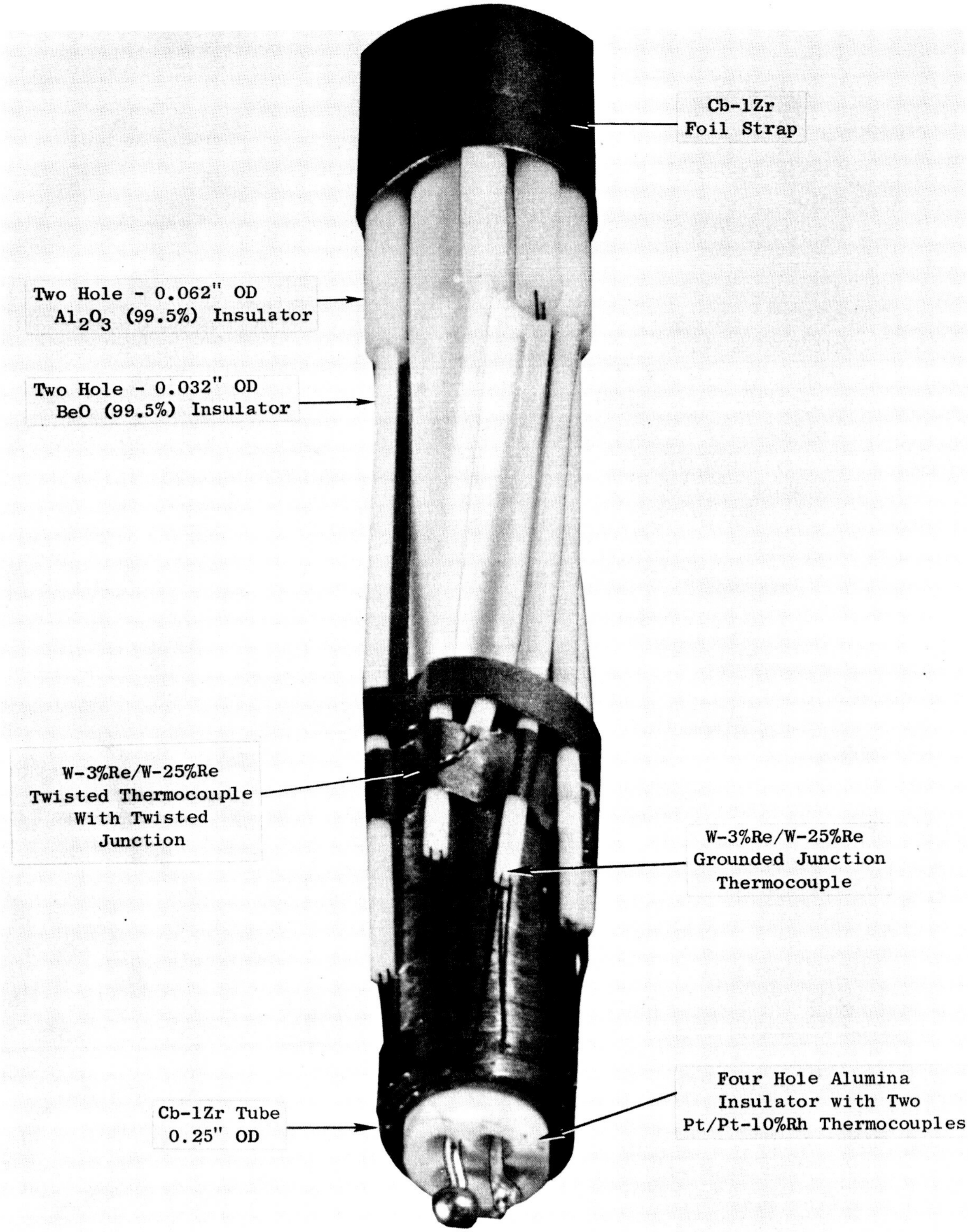


Figure 38. Thermocouple Bundle Used in Calibration of W-3%Re/W-25%Re Thermocouple Wire for the Prototype Corrosion Loop. (Orig. C65031521).

inch 99.5% BeO was added to eliminate contact and possible reaction between Cb-1Zr and alumina. Four such thermocouples were made from consecutive lengths of wire to be used for instrumenting the Prototype Loop. These individual thermocouple assemblies with four other samples of different wire were attached to the insulator containing the reference thermocouples by means of Cb-1Zr foil straps. The entire assembly was made rigid to eliminate the possibility of slippage between various parts. Three of the hot junctions were formed by resistance welding the individual alloy wires to the Cb-1Zr tube in a manner similar to that to be used in the actual loop application. A copper alloy tip was used with the spot welder and the weld area was flooded with argon during the welding procedure. One hot junction was formed by twisting the alloy wires together with no spot weld. Both types of junctions are evident in Figure 38.

The assembly described above was inserted in the calibration furnace as a single unit and strapped down to the chamber structure so that none of the thermocouple wires was in contact with the isothermal block. Copper leads from the vacuum feedthroughs were joined to the alloy wires by twisting both wires together. A copper-constantan thermocouple junction was located in the extension tube to measure the actual cold junction temperature.

Data were recorded by manually selecting the individual thermocouple channels, balancing the readout instrument, and recording the data on a log sheet. This process required approximately 2 minutes to complete and it was necessary to maintain stable conditions during that time period. Proof of stability was achieved by reading the reference thermocouple first, then the test thermocouples, and then a reference thermocouple again. When a change of more than 1 degree F was detected between before and after reference thermocouple readings, another data point was taken at the same condition. At temperatures above 1300°F, optical pyrometer readings were also taken. These required longer time periods and, in cases where conditions had changed slightly, the reference thermocouples were read a third time to coincide with the optical reading.

Calibration data were taken during three temperature cycles. During these runs, the pressure in the calibration furnace varied between a low of 2 x 10^{-8} torr at 400°F and a high of 6 x 10^{-6} torr at 2400°F. Two of the four thermocouples open circuited during the first heat-up due to a break in the W-3%Re leg at the hot junction. Therefore, all the data to be presented were obtained with two thermocouples, one with the grounded junction configuration and one with the twisted isolated junction configuration. Some difficulty was experienced during the calibration runs in maintaining the cold junction at the ice point due to thermal conduction along the alumina insulator. This problem will be eliminated in future calibration runs by using a number of short insulators in place of the continuous length used in this test. The fact that the cold junction temperatures were other than 32°F caused some problems in the data reduction area and made it necessary to obtain additional data covering the low temperature areas (up to 150°F).

Data were reduced by the following procedure.

Step 1. Cold junction temperature was converted from millivolts (copper constantan thermocouple) to degrees F.

Step 2. Block temperature was determined by adding an emf equivalent to the cold junction temperature to the reference thermocouple output and converting the sum to degrees F by means of National Bureau of Standards Bulletin 561. An average of the two Pt/Pt-10%Rh thermocouple readings (never more than 2.8°F of difference) was used as the actual block temperature.

Step 3. A table of output values for each thermocouple being calibrated was prepared from test data in the range 32° - 140°F.

Step 4. The millivolt signal equivalent to the reference junction temperature as defined in Step 1 above was calculated for each thermocouple being calibrated by linear interpolation of the table defined as Step 3.

Step 5. A corrected output signal was calculated by adding the millivolt signal determined in Step 4 above to the measured value. This information was used to generate a characteristic curve of corrected output millivolts vs the actual block temperature determined in Step 2 above.

Step 6. In order to facilitate curve plotting and permit use of an expanded scale, a straight line defined by coordinates (0, 0) and (24, 2400) where the first number is corrected millivolt output signal and the second is temperature in °F was used as a base line, and the difference between this line and the corrected output millivolts was calculated. The difference is plotted vs temperature as Figure 39.

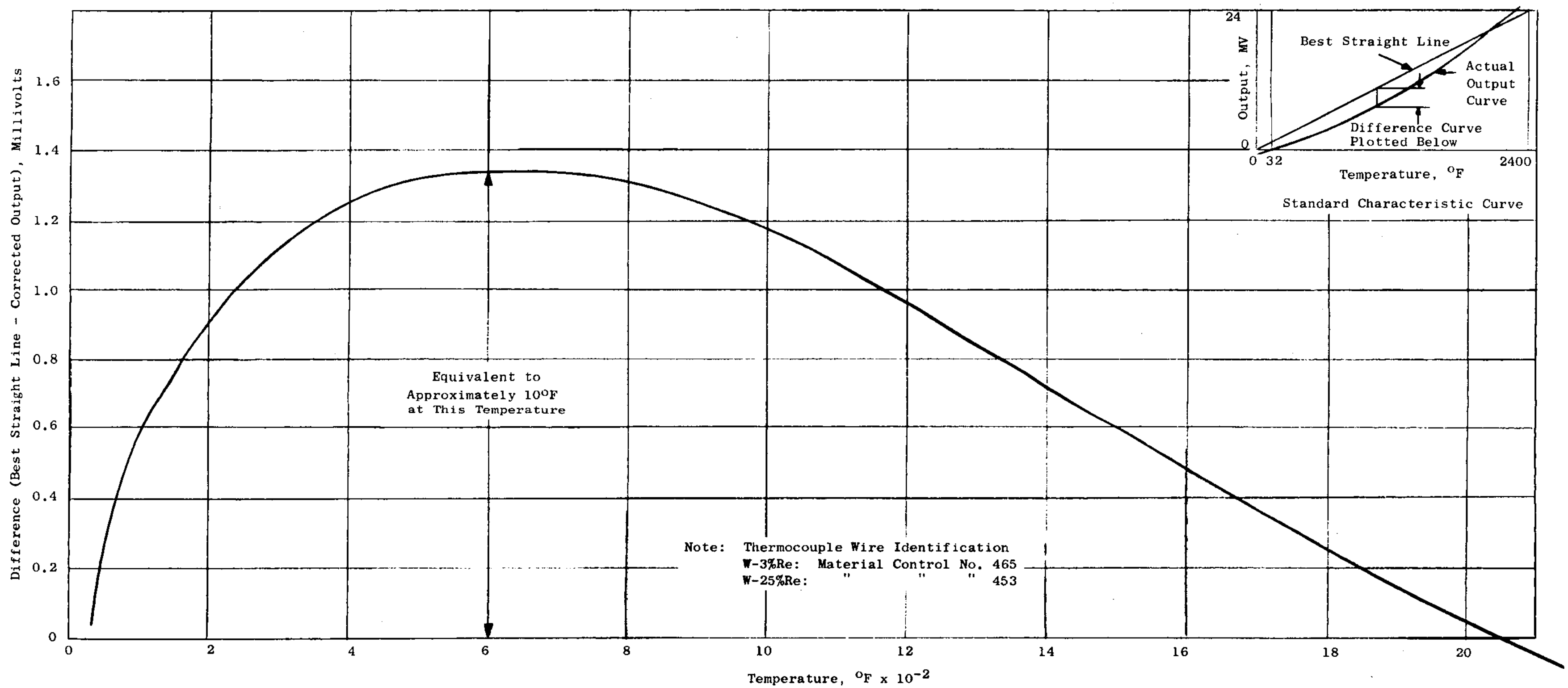


Figure 39. Difference Curve Vs. Temperature Obtained in Calibration of Two W-3%Re/W-25%Re Thermocouples Made from Wire Used to Instrument the Prototype Corrosion Loop.

The curve shown in Figure 39 is an average of all data points taken during three temperature cycles over the range from 80° to 2400°F for two thermocouples with different hot junction configurations but from the same spools of alloy wire. All but 8 of 35 stable points fall within 4°F of this line. Maximum deivation of any stable point from the line (up to 2200°F) is 9°F. The actual values used to define the curve are listed in Table IX. These are not actual test points but were picked off of the best line through the test data so that linear interpolation between any two points would cause a maximum error of 0.5°F.

A comparison between the two reference thermocouples and the optical pyrometer readings are shown in Table X. Optical readings were taken from 17 inches looking through a 1.5-inch high vacuum viewport at the bottom of the 0.5-inch diameter, 7-inch deep hole in the isothermal block. Correction factors based on calibration procedures were applied to the raw optical readings for the vacuum viewport window and the pyrometer lens. The generally good agreement is an indication that the block temperature was reasonably uniform and that all factors used to make the temperature measurements were consistent.

3. Calibration of Flowmeter Magnets

The permanent magnets of the flowmeters for the primary and secondary circuits of the Prototype Corrosion Loop have been stabilized and calibrated by repeated thermal treatments and flux measurements over the temperature range from room temperature to 900°F. The flux measurements obtained are plotted in Figure 40. These values were obtained for each magnet in two calibrations over the entire temperature range. Each calibration cycle was completed in approximately three hours and during each cycle the magnet was held at 900°F for 30 minutes. The decrease in the room temperature flux density of the magnets as a result of heating to 900°F was only 2% less than the original values as compared to the 10% decrease in the flux density of the Loop II flowmeter magnet prior to the loop test, as described in Section III.A.6 above.

4. Stress Diaphragm Pressure Transducer Calibration

The transducer for the Prototype Loop system was assembled, leak tested, and run through a preliminary calibration prior to attachment to the loop piping system. The preliminary calibration was conducted with the transducer connected to a Consolidated Controls Corporation excitation circuit. The transducer was pressurized with argon gas several times over the full operating pressure range (0 to 150 psig) and demonstrated a nearly linear output characteristics indicating 42 millivolts at 150 psi. Zero shift over seven full range pressure cycles was 1% of full-scale output. Zero shift over the last three cycles was 0.2% of full-scale output. The results are in good agreement with the deflection

TABLE IX. CHARACTERISTIC CURVE FOR W-3%Re/W-25%Re BASED ON VACUUM CALIBRATION DATA

Temp. °F	Millivolts Below Best[1] Straight Line	Millivolts Above Best[1] Straight Line	Total Output in Millivolts with 32°F Reference Junction
32.0	0.320		0
100.0	0.587		0.413
160.0	0.790		0.810
200	0.908		1.092
250	1.028		1.472
300	1.126		1.874
350	1.196		2.304
400	1.253		2.747
450	1.298		3.202
480	1.315		3.485
510	1.324		3.776
610	1.338		4.762
670	1.339		5.361
730	1.330		5.970
790	1.313		6.587
830	1.296		7.004
900	1.251		7.749
950	1.215		8.285
1000	1.173		8.827
1100	1.073		9.927
1200	0.961		11.039
1300	0.840		12.160
1450	0.662		13.838
1650	0.416		16.084
1750	0.300		17.200
1850	0.193		18.307
1950	0.092		19.408
2050	0		20.500
2150	--	0.086	21.586
2250	--	0.167	22.667
2350	--	0.240	23.740

[1] Defined by coordinates (millivolts, temperature), (0, 0) (24, 2400).

TABLE X. COMPARISON OF REFERENCE THERMOCOUPLES AND OPTICAL PYROMETER READINGS DURING THERMOCOUPLE WIRE CALIBRATION IN VACUUM

	Thermocouples			Optical Pyrometer[2]			
Reading No.	A[1] °F	B[1] °F	Average Thermocouple Indication °F	Corrected Temp. °F	Sight Glass Correction °F	Lens Corrected °F	Raw Optical Reading °F
26	1542.3	1544	1543.15	1549	+ 8.5	-10[3]	1550
27	1734.3	1731.6	1732.95	1726	+ 9.4	- 3	1720
29	1935.8	1933.3	1934.55	1915	+10.4	+ 5	1900
30	2031.9	2029.1	2030.5	2030	+10.8	+ 9	2010
31	2180.1	2177.9	2179.0	2182	+13.0	+14	2155
32	2338.2	2336.4	2337.3	2340	+15.3	+15	2310
33	2217.8	2215.2	2216.5	2209	+13.6	+15	2190
40	1622.1	1620.2	1621.15	1612	+ 9	- 7	1610
41	1794.8	1792.9	1793.85	1795	+ 9.8	0	1785
42	1997	1995	1996	1999	+10.7	+ 8	1980
43	2319	---	2319	2316	+15	+16	2285

1 Pt/Pt-10%Rh thermocouples, 0.20-inch diameter, in 99.0% Al_2O_3

2 Pyro Micro Optical Pyrometer, Serial No. M5344, Pyro Instrument

3 Extrapolated from available data.

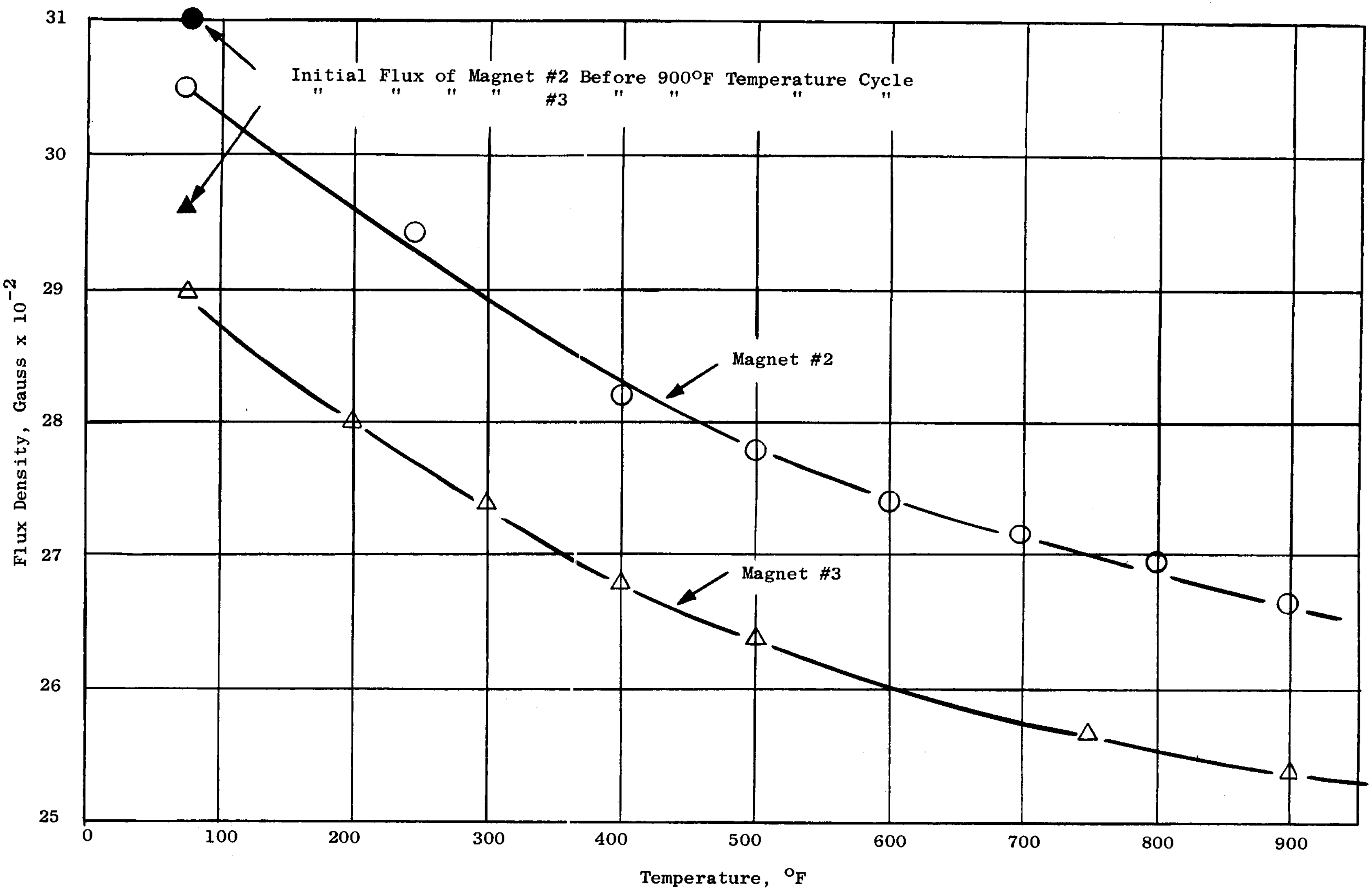


Figure 40. Magnet Flux Density Vs. Temperature of the Flowmeter Magnets of the Prototype Corrosion Loop.

test results obtained by Consolidated Controls Corporation on the diaphragm before it was joined to the transducer housing. They found the deflection of the diaphragm to be nearly linear over the range 0 to 150 psig, with a total deflection of 11.3 mils, or approximately 0.065 mils/psi.

The tests cited above were conducted at ambient temperature and it is expected that operation at elevated temperature will produce changes in both zero and span. However, compared to the Loop II transducer, this transducer showed a considerable improvement in linearity over the entire operating pressure range with a significant increase in sensitivity at pressures greater than 100 psi.

5. Tubing Stresses Due to Motor Torsion of the Helical Induction EM Pumps

The sodium pump which will be used in the primary circuit of the Prototype Loop and the pump used in the Loop II test exerts a calculated motor torque of 125 in-lbs at the rated power of 14 KW. The pump duct is inserted in an insulating can which also serves as a vacuum jacket to protect the Cb-1Zr alloy from oxidation at operating temperatures. The pump duct is not fastened or retrained in the insulating can to permit unrestrained thermal expansion of the duct.

An analysis of the stresses in the pump inlet and discharge tubes of the sodium circuit indicated that some form of restraint on the pump or the tubes was required to resist the motor torque of the pump during long-term operation at 2000°F. Assuming that restraining brackets on the tubes are located 10 inches from the pump face, then the calculated bending and torsional stresses in the pump tubes at rated power are as follows:

	Bending Stress	Torsional Stress
Inlet Tube 3/4-Inch Schedule 80	396 psi	595 psi
Discharge Tube 3/8-Inch Schedule 80	157 psi	440 psi

A similar calculation for the potassium pump of the Prototype Loop showed that a motor torque of 18.7 in-lbs would be exerted on the duct at the rated power of 3 KW. The calculated bending and torsional stresses in the tube with the restraining brackets located 10 inches from the duct are as follows:

	Bending Stress	Torsional Stress
Inlet Tube 0.375-Inch OD x 0.065-Inch Wall Tube Attached at 1.24 Inches from Centerline of Duct	970 psi	1050 psi
Discharge Tube 0.375-Inch OD x 0.065-Inch Wall Tube Attached at 0.77 Inch from Centerline of Duct	600 psi	1050 psi

In order to eliminate the possibility of tube distortion due to the motor torque of the pumps, support brackets of Cb-1Zr alloy have been located 5-1/2 inches from both of the pump faces. This reduces the tubing stresses to insignificant levels.

6. Temperature Limitation of Cb-1Zr Alloy Valve Bellows

An analysis has been performed to estimate the temperature limitation of Cb-1Zr alloy bellows of the type currently being used in this program. The bellows are formed* hydraulically in two steps with an intermediate recrystallization anneal to the following general specifications:

OD:	0.500 Inch
ID:	0.375 Inch
Free Length:	1.125 $\pm$ 0.062 Inches
No. of Convolutions:	28
Travel:	0.125 Inch
Maximum Rated Internal Pressure:	3,600 psi

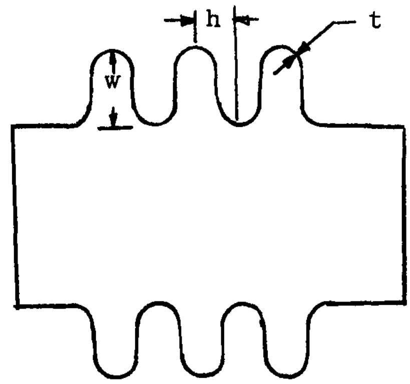
The dimensions of the bellows blanks prior to forming were 0.375-inch OD x 0.0080 $\pm$ 0.0005-inch wall x 7.0 inches long. Metallographic examination of a number of longitudinal sections of formed bellows revealed a minimum bellows wall thickness of 0.006 inch, and this dimension was used in estimating the temperature limitation of the Cb-1Zr alloy bellows.

---

* Standard Thomson Company, Waltham, Massachusetts.

Operating Stress - The range of stress in the bellows during loop operation can be approximated by:

$$S_R = \frac{1.5\ E\ t\ \Delta}{h^{0.5}\, w^{1.5}\, Nd} + \frac{P\ w^2}{2t^2} \qquad (14)$$



where

S_R = range of stress due to expansion and pressure, psi

E = modulus of elasticity, psi

t = bellows thickness, inch

Δ = total movement range, extension or compression, inch

h = pitch of half-corrugation, inch

w = height of corrugation, inch

Nd = number of half corrugation

P = internal pressure, psi

The equation for the stress range consists of two terms: the first term reflects the stress due to the bellows travel and the second term refers to the stress due to internal pressure. For a deflection of 0.100 inch at 150 psi internal pressure; the bellows stresses are approximately 80,000 psi for the deflection term and 11,700 psi due to the internal pressure. Using estimated minimum stress-rupture properties of Cb-1Zr for 10,000-hour life, the temperature limit placed upon the valve bellows would be approximately 1400°F without making any allowance for a design safety factor.

Spring Rate - The axial deflection of the Cb-1Zr bellows as a function of load was experimentally measured and is presented in Figure 41. The bellows spring rate at room temperature is 118 lbs per inch. The force required to deflect the bellows 0.10 inch, which is the normal travel range of the metering valve, is 11.8 lbs compared to a calculated value of 14.6 lbs based on the equation:

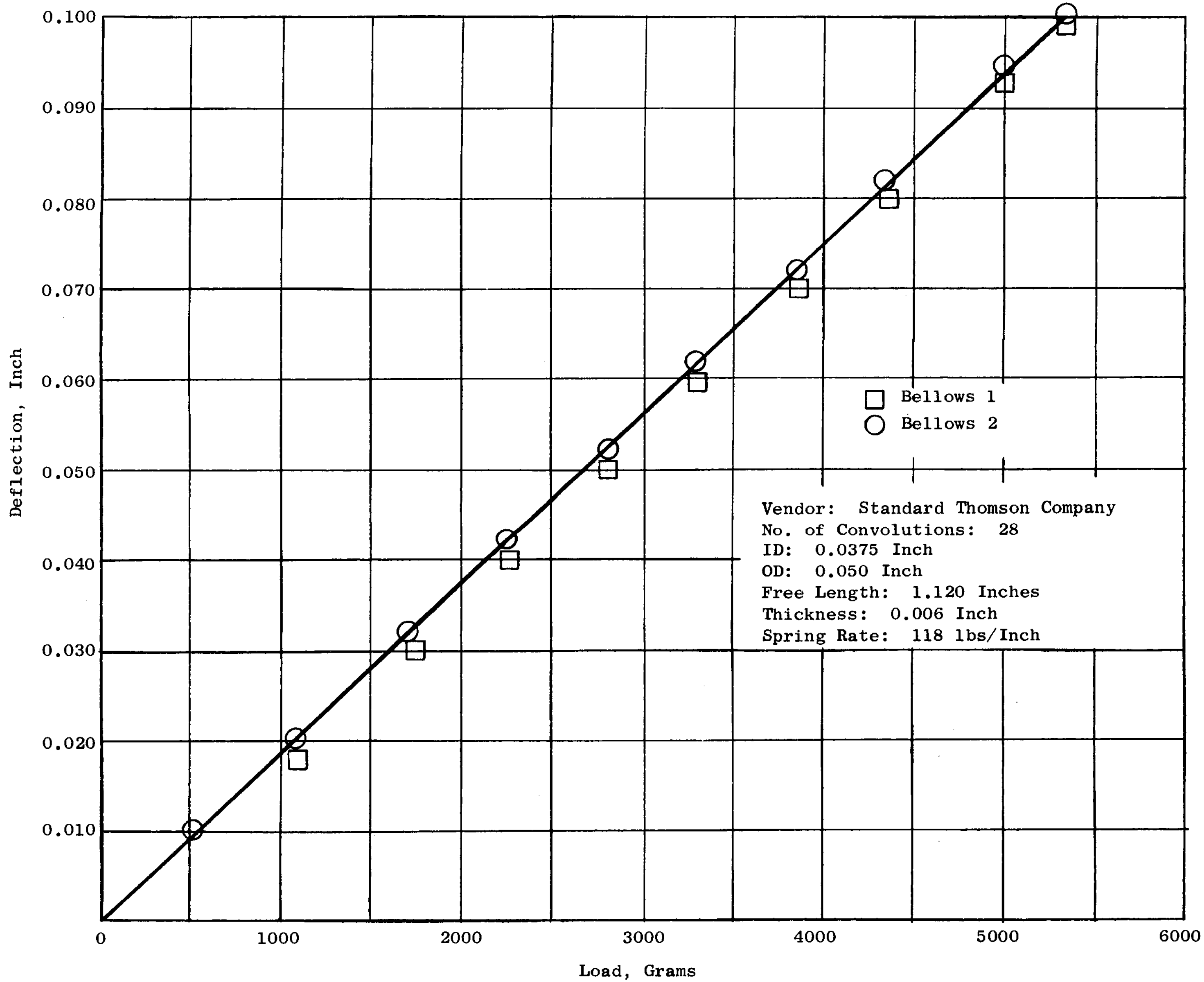


Figure 41. Deflection Vs. Load for the Prototype Corrosion Loop Cb-1Zr Alloy Valve Bellows.

$$F = \frac{4 E D t^3}{3 h^{0.5} w^{2.5} N d} \quad (14)$$

where

D = mean bellows diameter, inch

E. Prototype Corrosion Loop Operation

Operation of the Prototype Loop is scheduled for July, 1965 and a test plan for this experiment has been submitted to NASA for approval. The Table of Contents of this test plan is listed below:

CONTENTS

I TEST OBJECTIVE

II GENERAL DESCRIPTION OF THE TEST SYSTEM AND THE TEST CONDITIONS

III LIST OF DRAWINGS

IV DESCRIPTION OF PRINCIPAL LOOP COMPONENTS

V LOOP INSTRUMENTATION AND INSTALLATION

VI SEALING, BAKEOUT AND LEAK CHECKING OF THE TEST CHAMBER

VII PURIFICATION AND LOOP FILLING PROCEDURE

VIII CHECKOUT OF SAFETY CIRCUITS

IX PRETEST LOOP CHECKOUT

X CALIBRATION OF LOOP INSTRUMENTATION

XI TEST OPERATION

XII EVALUATION OF COMPONENT PERFORMANCE

APPENDIX I - List of Drawings - Prototype Corrosion Loop

APPENDIX II - List of Drawings - Alkali Metal Purification, Handling and Sampling System for the Prototype Corrosion Loop

The proposed Test Operation portion, Section XI, of the Test Plan is included below with a schematic diagram of the loop, Figure 42, and the instrumentation drawing, Figure 43, showing the location of thermocouples on various loop components.

XI TEST OPERATION

A. Recording of Test Data

The test operator will maintain an official log book of all pertinent test data, observations and test progress to present a complete but concise history of the loop operation. In addition to the log book, he will record daily all test data on a standard test data sheet maintained in chronological order in a loose leaf notebook. A partial pressure gas analysis of the residual gases in the vacuum chamber will be recorded daily.

B. Operating Instructions

The project engineer will instruct and qualify all loop operators in the control and operation of the loop and auxiliary test equipment. In addition to these instructions, a complete file of loop drawings and equipment manuals will be available to the operator at the test site. A thorough review of the safety and protection circuits of the loop will be made during the operation of the test. No safety circuit will be bypassed without written permission in the log book by the project engineer or project manager.

C. Test Startup

The loop and vacuum chamber have previously completed an initial bakeout and are currently at room temperature at a total pressure less than 1×10^{-8} torr. During the following operation the rate of increasing the loop temperature will be controlled by the outassing rate of the loop components as they exceed the bakeout temperature. The permissible vacuum chamber pressures as a function of temperature are listed at the end of this section (C.22).

The potassium and sodium inventory are solidified in their respective surge tanks under vacuum. The following procedures will be used to fill the primary (sodium) and secondary (potassium) loops and to bring the loop to the design operating conditions.

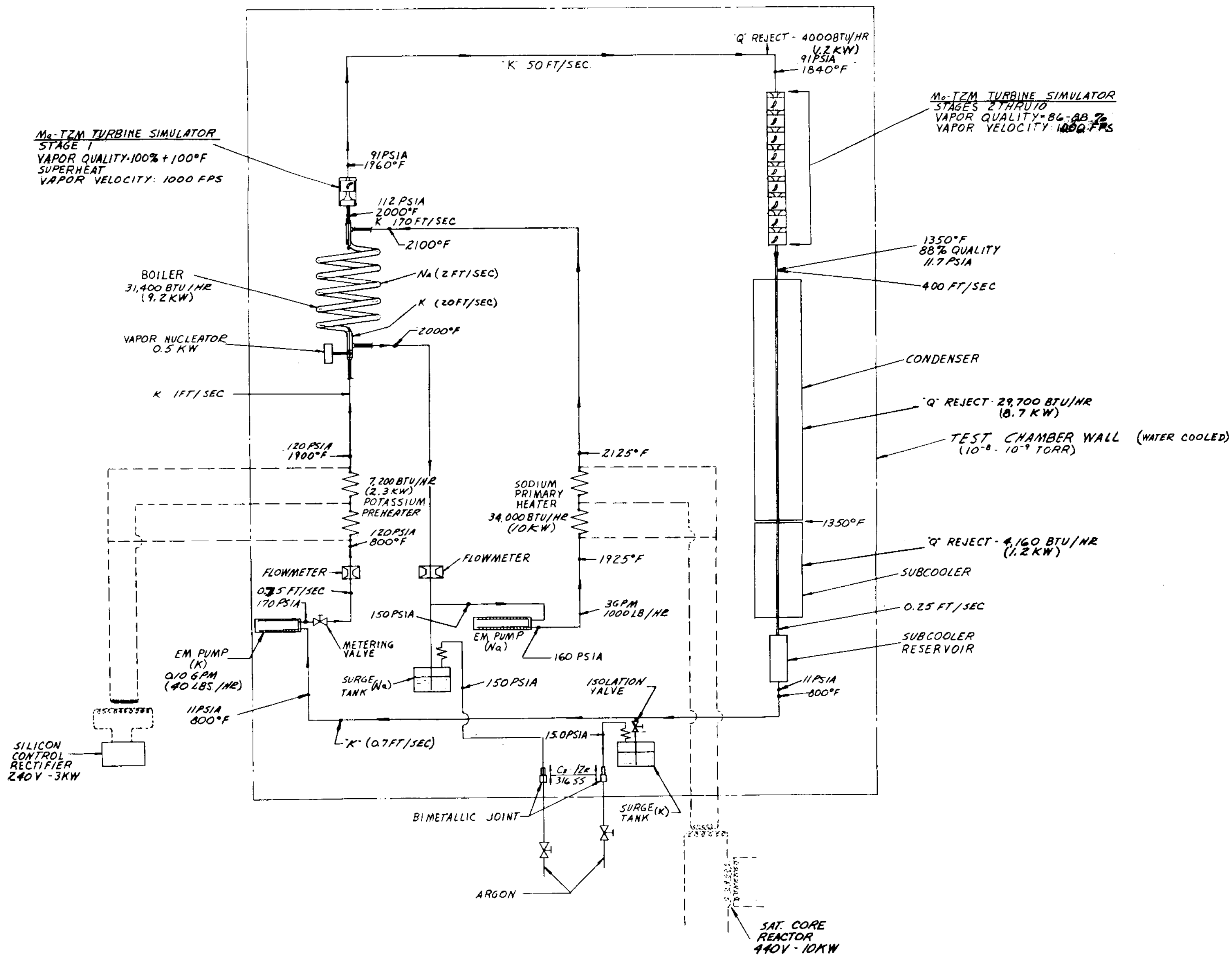


Figure 42. Schematic Diagram of the Prototype Corrosion Loop.

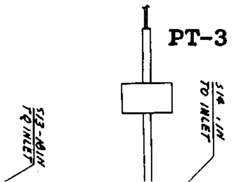
NOTE:

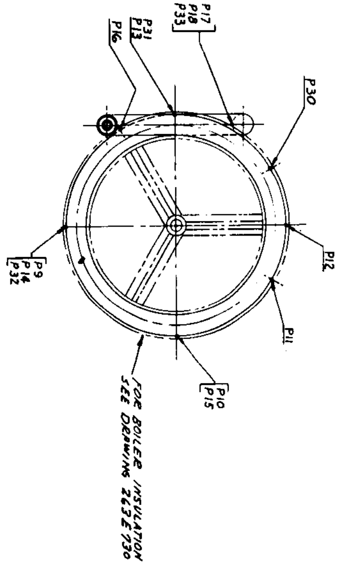
P - Indicates primary (sodium) circuit thermocouple.

S - Indicates secondary (primary)

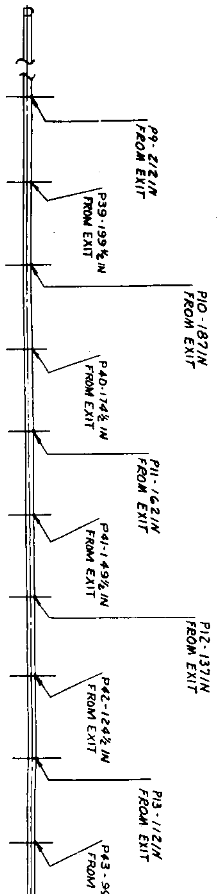
PT-1 thru PT-4 - Slack Diaphragm Pressure Transducers

PT-5 - Stressed Diaphragm Pressure Transducer





VIEW AT A-A



BOILER SECTION (DEVELOPED)

Fi

1

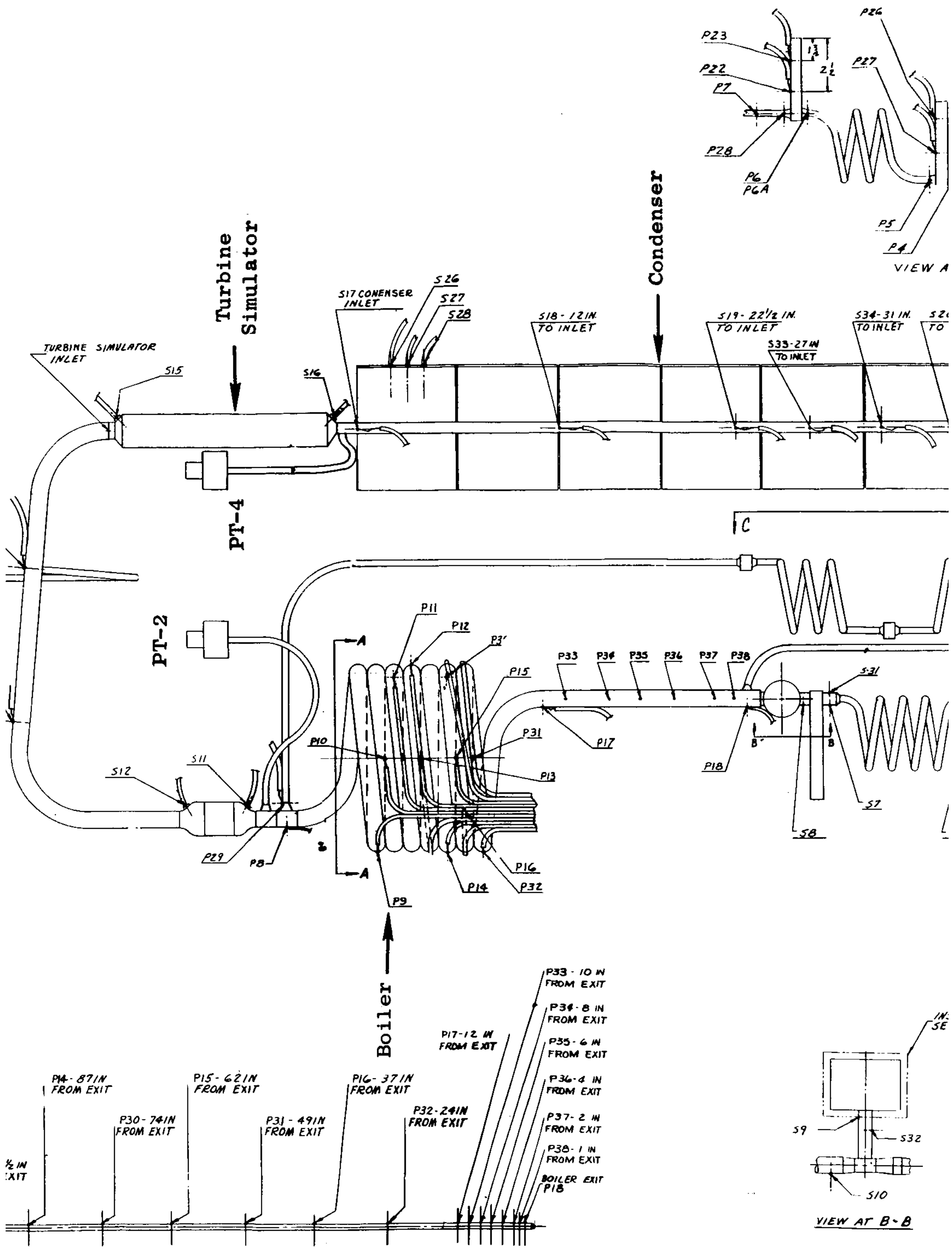
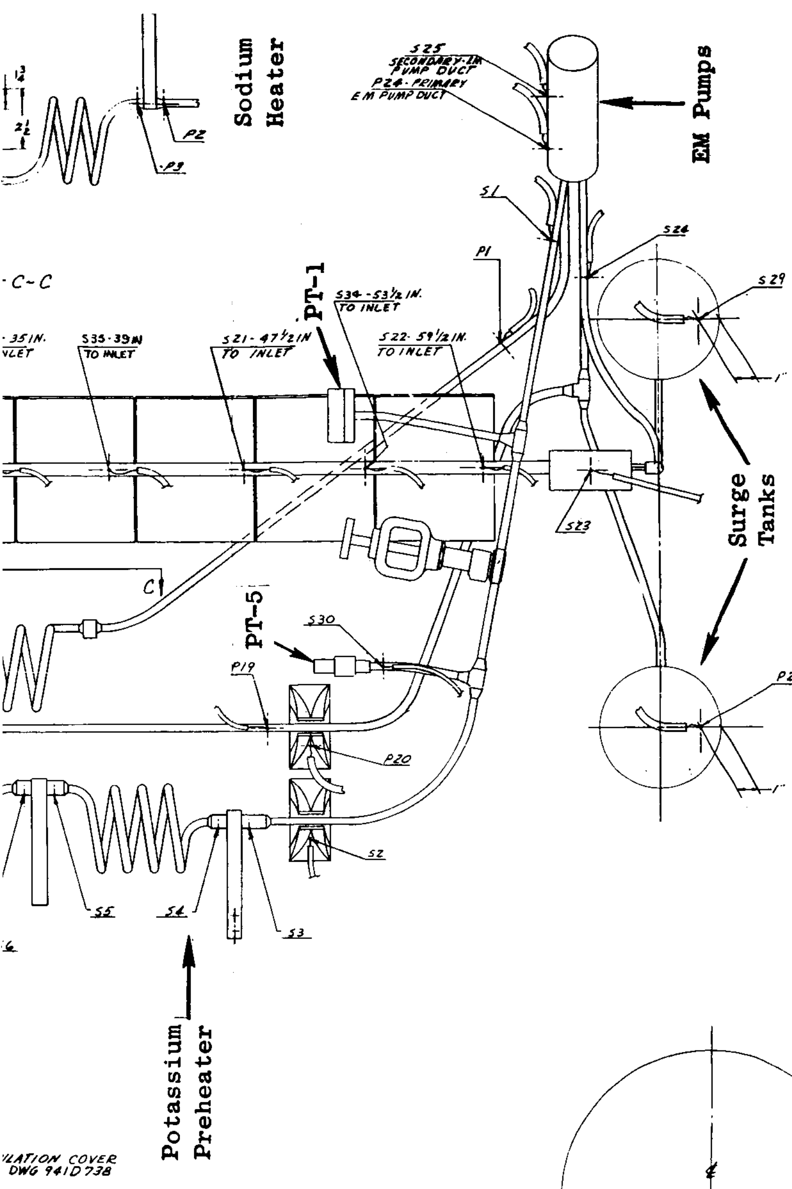
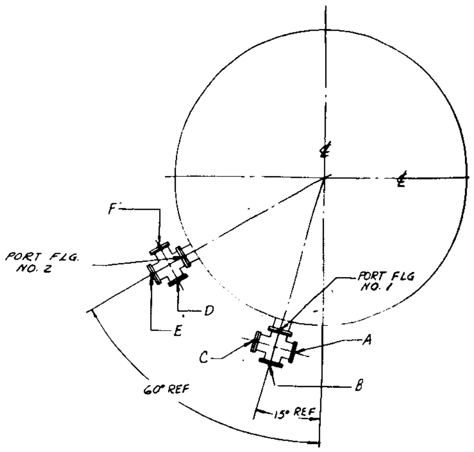
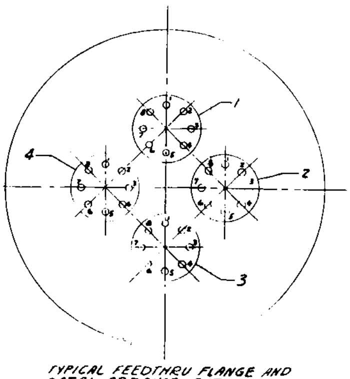


Figure 43. Prototype Corrosion Loop Thermocouple Instrumentation Layout.





FEEDTHRU CHAMBER WALL
VIEW AT LOWER TANK
NO SCALE



TYPICAL FEEDTHRU FLANGE AND
OCTAL ARRANGEMENT
REF. A, B, C, D, E & F

3

1. Turn on the vacuum chamber electrical heaters for 500°F bakeout of bell jar, sump and spool section.

2. Turn primary and secondary EM pump power to 10% of full power and allow the pump temperature to increase to 500°F and then adjust the power to maintain 500°F.

3. To fill the primary loop, check the surge tank temperature and if the temperature is greater than 250°F, increase the argon pressure to 150 psia.

4. The primary loop will fill automatically as the sodium is forced out of the surge tank into the loop.

5. The fill will be detected by the indicated flow on the flowmeter recorder and temperature changes in the primary circuit.

6. Before filling the secondary loop, open the isolation valve.

7. Close the metering valve with 1 ft-lb of torque.

8. Slowly increase the argon pressure of the primary loop to 2.0 psia which is equivalent to a 70-inch column of potassium above the surge tank (0.0283 lbs/sq in/in of potassium at 480°F).

9. Verify the height of the rising potassium in the condenser by the change in temperature of thermocouples on condenser/subcooler pipe. Temperature change at T/C S-17 (condenser inlet) indicates K at proper height. No change in temperature should be detected in the preheater side of the loop.

10. Close the isolation valve with 2 ft-lbs of torque.

11. Increase the secondary gas pressure to 100 psia in the surge tank and check for internal leak across the isolation valve seat by a pressure change on the pressure transducer PT-1.

12. If no leak is detected after one hour reduce the argon pressure to 15 psia.

13. Boiling operation can now be started.

14. Slowly increase the temperature of the primary circuit to 1350°F on manual control with flow rate at 2 gpm.

15. Check the power setting of the secondary EM pump and adjust to 10%.

16. Slowly open the metering valve to 40-45° rotation which will be equivalent to a 20 to 40 psi drop at design.

17. Boiling and condensing in the secondary loop will begin with this operation. Maintain flow and power input conditions until thermal equilibrium is achieved in secondary circuit.

18. Check the potassium vapor quality at the boiler exit.

    a. If quality is 100% or less decrease the potassium flow rate to obtain 25° to 75°F of superheat in potassium vapor.

    b. If vapor is superheated slowly increase the potassium flow rate while maintaining 25° to 75°F of superheat.

19. Slowly increase the temperature of the primary loop in 25°F increments.

20. Turn power on potassium preheater and adjust power to obtain a boiler inlet temperature 25°F below the potassium saturation temperature.

21. Continue to increase the primary loop temperature, the secondary flow rate and the preheater temperature until the following test conditions are reached:

Boiling Temperature	-	1900° ± 25°F
Superheat Temperature	-	2000° ± 10°F
Condensing Temperature	-	1350° ± 25°F
Potassium Flow Rate	-	35 lb/hr ± 5 lb/hr

Nominal values of pressures, temperature, flow velocities, and power inputs to various loop components are given in Figure 2 (Figure 42 of this report).

22. The maximum test chamber pressures which shall not be exceeded during test startup and operation are given below:

Test Period	Maximum Loop Temp., °F	Maximum Ionization Gauge Pressure Torr
Bakeout	500	5×10^{-5}
Test Startup	1000	1×10^{-5}
Test Startup	1500	5×10^{-7}
At Test Conditions		
0-100 Hrs.	2125	1×10^{-7}
100-500 Hrs.	2125	5×10^{-8}
500-2,500 Hrs.	2125	1×10^{-8}

In the event that the ionization gauge pressure limitations given for the loop operation are exceeded a partial pressure scan will be obtained to determine the concentration of reactive gases. These results will be reported to the project engineer immediately to obtain approval to continue the test.

23. Continue test at above conditions for 2,500 hours.

24. The effect of a change in the potassium flow, temperature, or inventory are summarized in Table I (Table XI of this report).

D. Loop Shutdown Procedures

1. Normal - The following procedure will be used when a scheduled loop shutdown has been requested by either the project engineer or project manager for either loop or facilities maintenances which will prevent the normal operation of the loop. The shutdown procedure will normally require one hour and all adjustments will be made in small, carefully controlled increments to avoid liquid carryover to the turbine simulator.

TABLE XI. PROTOTYPE LOOP OPERATION

Operation	Results of Operation: Pressure Changes	Results of Operation: Temperature Changes
1. Increase potassium flow rate Quality 100%	Pressure drop increases across turbine simulator	Condenser temperature decreases; boiling temperature increases
2. Decrease potassium flow rate Quality 100%	Pressure drop decrease across turbine simulator	Condenser temperature increases; boiling temperature decreases
3. Increase sodium temperature Flow rate constant Quality 100%	Pressure drop increases across turbine simulator	Boiler temperature increases; condenser temperature decreases
4. Decrease sodium temperature Flow rate constant Quality 100%	Pressure drop decreases across turbine simulator	Boiler temperature decreases; condenser temperature increases
5. Increase potassium inventory Flow rate constant Quality 100%	Loop pressure increases and pressure drop across turbine simulator decreases	Boiler temperature increases; condenser temperature increases
6. Decrease potassium inventory Flow rate constant Quality 100%	Loop pressure decreases and pressure drop across turbine simulator increases	Boiler temperature decreases; condenser temperature decreases

a. In sequence, decrease:

   i. Secondary pump power

   ii. Preheater power

   iii. Heater power

   iv. Primary pump power

b. When the temperature of the loop is less than 1000°F, turn off all loop power.

c. Evacuate primary and secondary surge tank to 0 psia.

d. Primary circuit will dump automatically; do not dump secondary circuit unless it is requested by either the project manager or project engineer.

2. Emergency - The following procedure will be used only in event of:

   Severe leak in the loop.

   Loss of electrical power or water supply, which will prevent normal loop operation.

   Abnormal behavior of the loop including loop pressure or temperature excursions following establishment of steady state operation.

a. In sequence, without delay:

   i. Heater power off

   ii. Preheater power off

   iii. Primary and secondary pump power off

   iv. Evacuate primary and secondary surge tanks to 0 psia. Primary loop will be dumped automatically when primary surge tank is evacuated.

b. If leak is observed in the secondary system, open the isolation valve. Secondary system will then dump.

c. After secondary loop has been dumped, close the isolation valve with 2 ft-lbs of torque.

F. Refluxing Potassium Compatibility Tests

The two refluxing potssium capsule tests which are being conducted at 2000°F to determine the extent of mass transfer of Mo-TZM alloy tubular inserts in the condenser region of Cb-1Zr alloy capsules have been described in previous reports (15, 16). The 2,500-hour test was completed during the past quarter and preliminary examination of the test components has been completed. Following removal of the 2,500-hour test (capsule #1), the 5,000-hour test (capsule #2) was restarted and, as of April 15, 3,362 hours of operation had been completed.

The test chamber pressure during the 2,500-hour test of Capsule #1 may be summarized as follows:

Test Hours	Pressure, Torr
0-20	9×10^{-7} to 2×10^{-7}
20-100	2×10^{-7} to 1×10^{-7}
100-500	1×10^{-7} to 2×10^{-8}
500-2,500	2×10^{-8} to 6×10^{-9}
System at Room Temperature Following Test	8×10^{-10}

The potassium condensing rate for capsule #1 during the 2,500-hour test was calculated by means of the heat measurements obtained on the water-cooled heat exchanger which surrounded the condenser zone of the capsule as previously described (17). Failure of a thermistor used to measure the temperature rise of the water after 600 hours of testing prevented the determination of condensing rates for a large portion of the test. Measurements during the first 600 hours of the test yielded condensing rates which varied from a minimum of 28.9 $lbs/hr\ ft^2$ to a maximum of 29.3 $lbs/hr\ ft^2$. The defective thermistor was replaced at the end of the 2,500-hour test and a condensing rate of 31.5 $lbs/hr\ ft^2$ was measured. Additional comments on the condensing rate measurements are given below relevant to the capsule #2 test.

Following completion of the 2,500-hour test, capsule #1 was removed from the test chamber, and a preliminary evaluation was performed on the test components. Figure 44 shows the capsule following removal from the test chamber. The capsule was opened in the vacuum/purge welding chamber, and the boiler and condenser portions of the capsule were placed in the vacuum distillation unit

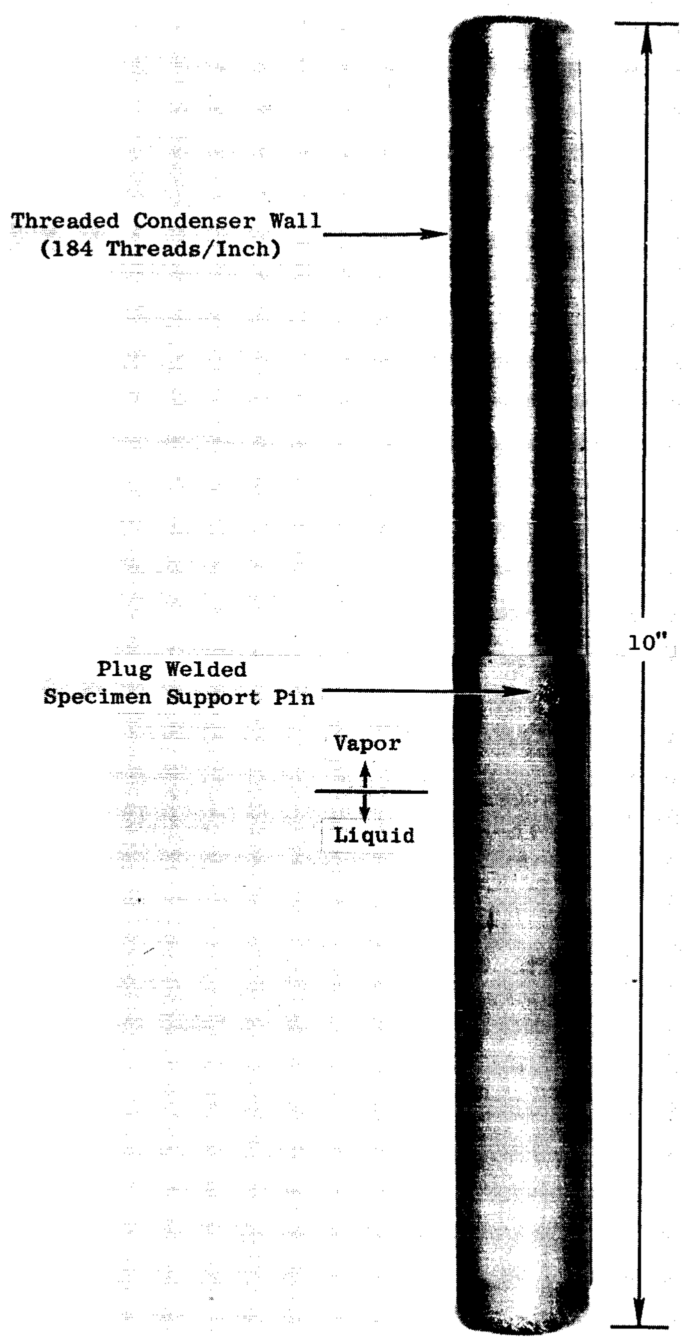


Figure 44. Cb-1Zr Alloy Refluxing Potassium Test Capsule Containing Mo-TZM Alloy Tubular Inserts in the Condenser Zone Following Completion of 2,500-Hour, 2000°F Test. (Orig. C65032521)

unit which has been described in an earlier report (18). The distillation unit was evacuated to 1 x 10^{-5} torr and heated to 875°F. Pumping was continued on the system for 4 hours at which time the evacuation line was valved shut. Distillation continued for 38 hours. The distillation unit was then allowed to cool to room temperature and subsequently was opened in the vacuum/purge welding chamber. All the potassium from the two ends of the capsule (21.8 gm total) had collected on the air cooled condenser and none was detected on the top portion of the capsule which contained the Mo-TZM alloy inserts or in the boiler region where most of the potassium was located prior to distillation.

Following removal from the welding chamber, the capsules were inverted and tapped gently to determine if any loose particulate material remained after distillation. None was found.

Following distillation, the five Mo-TZM alloy tubular inserts were removed from the condenser region of the capsule. All the Mo-TZM alloy specimens except the top one (specimen #1) were removed without difficulty. Specimen #1 was removed with some difficulty due to shrinkage of the Cb-1Zr pipe during EB welding of the top cap while sealing the capsule prior to testing.

Weight change data obtained on the five Mo-TZM alloy insert specimens located in the condenser region and the Cb-1Zr alloy sheet specimen from the boiler (liquid) region are given in Table XII. As may be noted in this table, the Mo-TZM alloy specimens experienced only minor weight changes during the 2,500-hour test. Mo-TZM specimen #1 showed a very slight weight gain and all or a portion of this gain is attributed to pickup of very small particles of Cb-1Zr from the test capsule in several areas where detectable bonding occurred between the insert and the capsule. The capsule after sectioning is shown in Figure 45. As may be noted in this photograph, the areas of the Cb-1Zr alloy which were in contact with liquid during the test were dark in appearance while the areas in contact with vapor retained a bright appearance. The reverse was true for the Mo-TZM alloy specimens. The insert specimens were darkened in the areas in contact with liquid (annulus between capsule and specimens) and bright in appearance on the ID surfaces which were in contact with condensing vapor. Tests will be conducted to determine the cause of the darkened appearance.

Additional chemical and metallographic examination of the specimens and capsule from this test will be performed following completion of the 5,000-hour test on capsule #2.

The condensing rate and temperature data which have been obtained on Capsule #2 during the first 3,300 hours of test operation are shown in Figure 46. In both this test and in capsule test #1 a definite drop in temperature with time has been observed with a constant voltage input to the heaters. This drop

TABLE XII. WEIGHT CHANGE DATA OBTAINED ON SPECIMENS FOLLOWING Cb-1Zr/Mo-TZM REFLUXING POTASSIUM CAPSULE TEST

Test Conditions:

Test Duration:	2,500 Hours
Capsule Temperature:	2000°F
Condensing Rate:	30.4 $lbs/ft^2/hr$ (0.248 $gm/cm^2/min$)

Specimen	Weight, gm		Weight Change	
	Before	After	mg	mg/cm^2[1]
Mo-TZM[2] #1 (Top of Condenser)	27.2833	27.2843	+1.0	+0.07
Mo-TZM #2	31.5581	31.5575	-0.6	-0.04
Mo-TZM #3	31.6653	31.6650	-0.3	-0.02
Mo-TZM #4	31.6128	31.6134	+0.6	+0.04
Mo-TZM #5 (Bottom of Condenser)	31.6507	31.6552	+4.5	+0.32
Cb-1Zr[2] (Boiler Region)	19.0209	19.0221	+1.2	+0.04

[1] Loss of 25.7 mg/cm^2 for Mo-TZM alloy is equivalent to 1 mil of uniform surface removal.

[2] Nominal dimension of Mo-TZM insert specimens (inches):

	Length	OD	Wall Thickness
Specimen #1	0.87	0.85	0.078
Specimens #2, #3, #4, #5	1.0	0.85	0.078

[3] Nominal dimensions of Cb-1Zr sheet specimen:

0.5-inch wide x 3-1/2 inches long x 0.0792-inch thick.

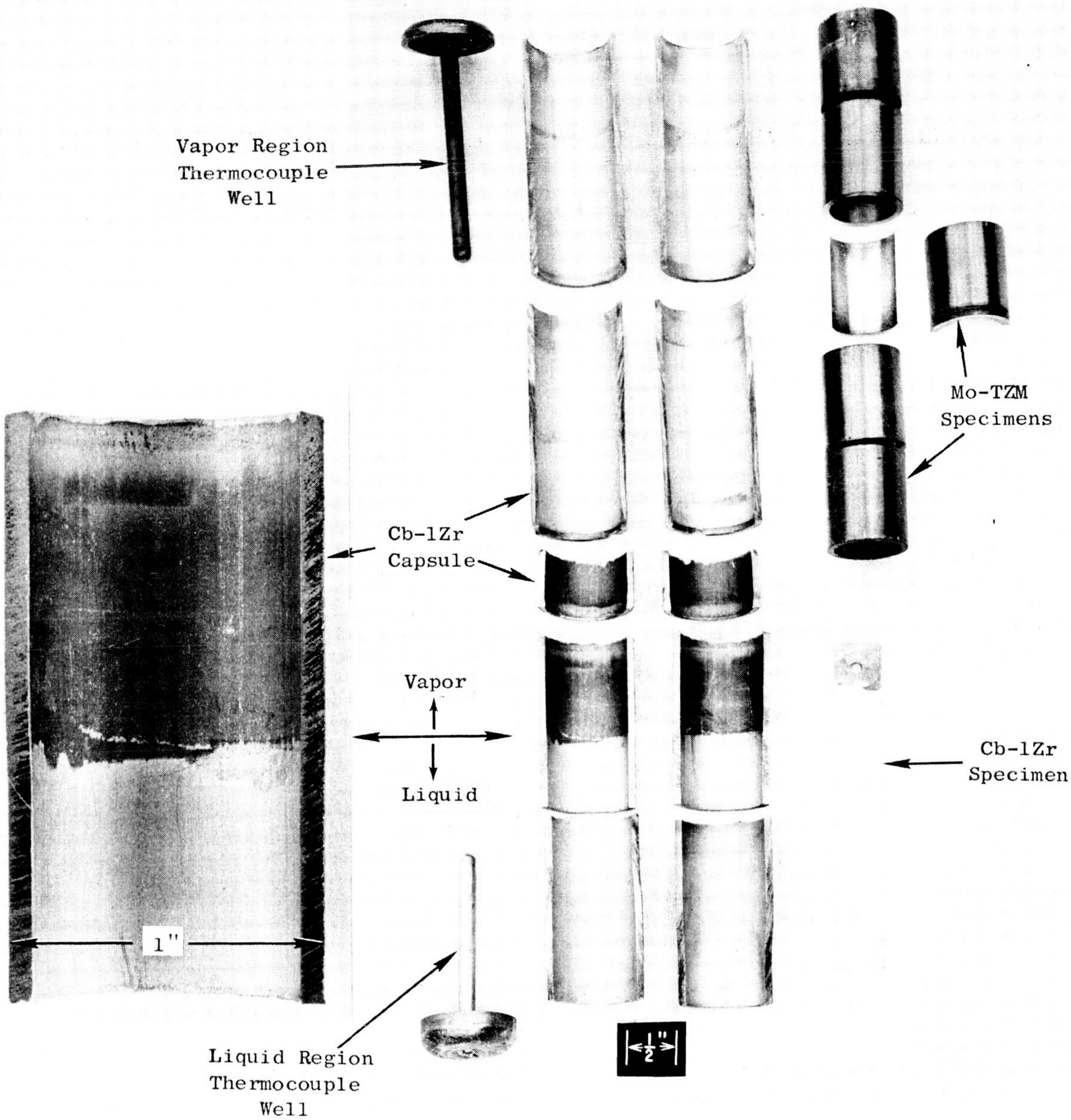


Figure 45. Potassium Reflux Capsule Components Following 2,500-Hour Test at 2000°F. Enlarged View of Cb-1Zr Capsule Wall at Liquid-Vapor Interface Shown on the Left.

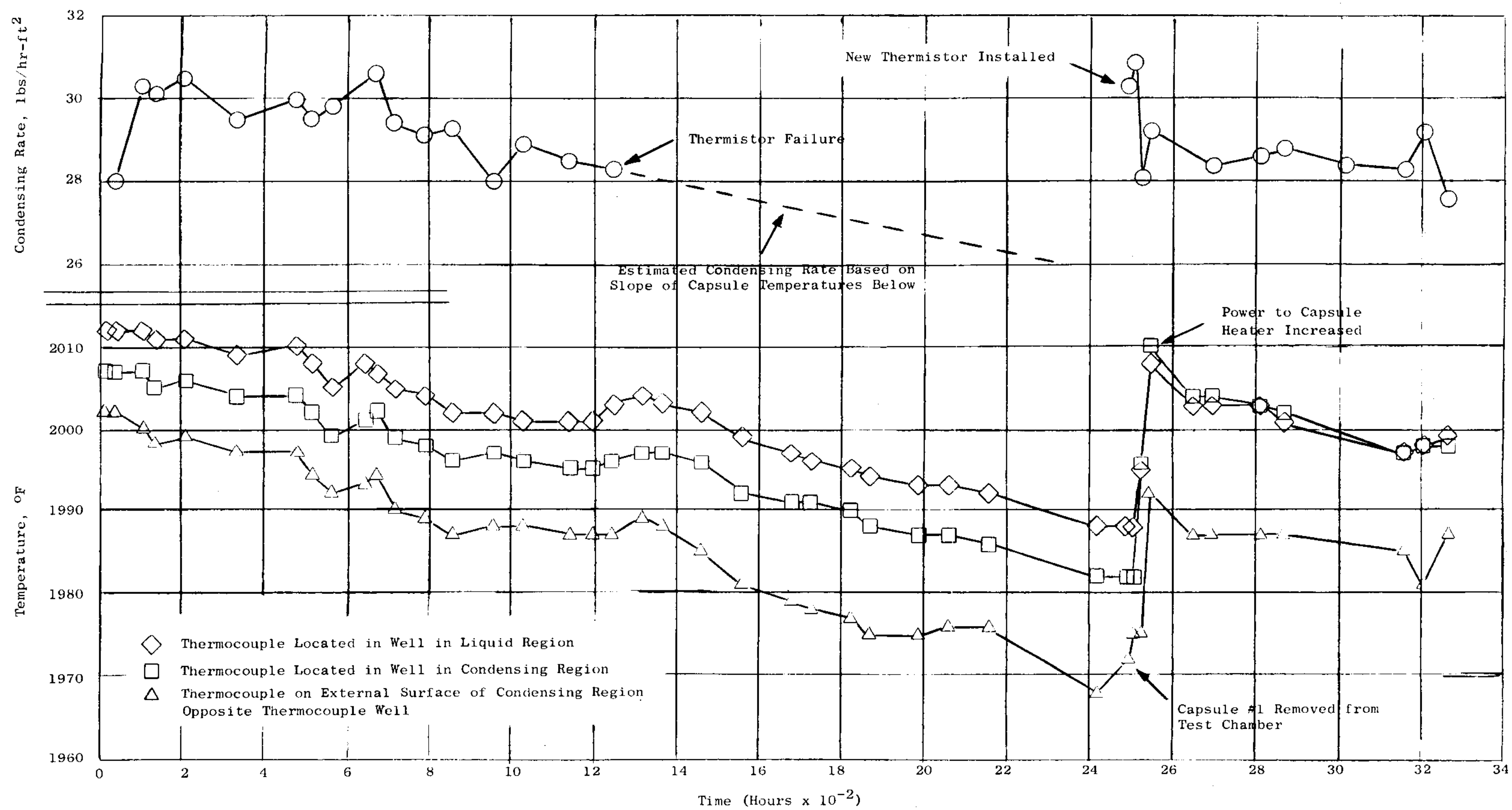


Figure 46. Condensing Rates and Temperatures of Mo-TZM Refluxing Potassium Capsule Test No. 2 During the First 3300 Hours of Operation.

in temperature is attributed to a decrease in the heater resistance. In capsule #2 the resistance of the heater at room temperature was found to have decreased about 3.2% after 2,500 hours of testing. A similar result was noted for capsule test #1. The drop in the temperature of capsule #2 is clearly reflected in the condensing rate decrease observed for this capsule.

The condensing rate during the first 3,300 hours of the test has varied from a minimum of 28 lbs/ft^2 hr to a maximum of 30.9 lbs/ft^2 hr. A heat of vaporization of 736.9 BTU/lb at 2000°F was used in making the condensing rate calculations (19). Calculations of the emittance of the threaded Cb-1Zr alloy condenser wall based on the measured heat rejection and system temperatures yielded a value of 0.23. This value is considerably less than was anticipated for this type of surface.

G. Helium Analysis System

The analysis system for the measurement of oxygen and moisture in the argon cover gas for the Prototype Loop has been received. This unit consists of a Beckman Model 27901 electrolytic hygrometer and a Beckman Model 89 trace oxygen analyzer with associated gas handling system. A photograph of this system is shown in Figure 47.

The electrolytic hygrometer has five ranges: 0-10, 0-30, 0-100, 0-300, and 0-1,000 ppm full scale. The oxygen analyzer has two ranges: 0-10 and 0-50 ppm full scale.

This analysis system has been temporarily attached to the exit line of the purification train used to purify the helium for welding. The system has been checked out and is now used routinely in place of the Brady apparatus and dewpoint cup to analyze the inlet helium to the welding chamber. Analyses have shown that the helium is of consistently high purity with typical values of 0.5 ppm oxygen now being obtained.

This analysis system will continue in use for helium analyses for the welding chamber until the time that it is needed for Prototype Corrosion Loop operation.

H. Weld Contamination Study

A study of contamination of welds on Cb-1Zr alloy sheet has been performed. Automatic fusion welds at 2 inches per minute were made on both 0.062-inch sheet and 0.125-inch sheet. A total of 16 weld specimens were prepared and analyzed for changes in O, N, and H by vacuum fusion analyses. Some Kjeldahl nitrogen analyses were also performed.

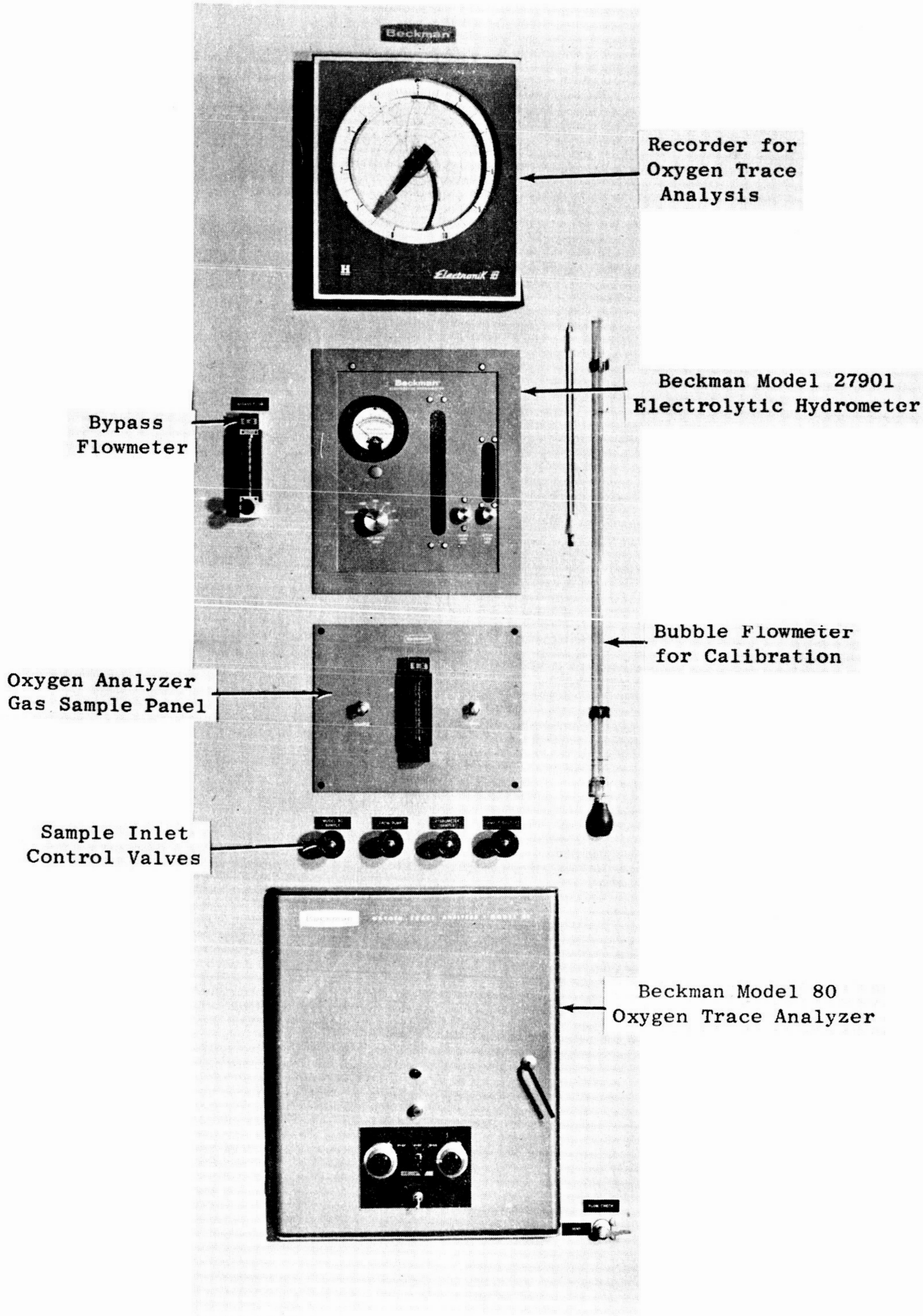


Figure 47. Beckman Electrolytic Hygrometer and Oxygen Trace Analyzer to be Used for Monitoring Purity of Argon Cover Gas for Prototype Corrosion Loop. (C65041409)

Air was added to the chamber from a gas burette. Welds were made at various chamber purity levels between 0 and 100 ppm air added. Quite good agreement was found between the air added and the analyses of the chamber atmosphere with the mass spectrometer. Welds were also made at moisture levels between 0 and 50 ppm. No significant increase in O, N, and H was found in any of the weld specimens. The details of this contamination study will be reported in a topical report entitled, "Purification and Analysis of Helium for the Welding Chamber" which will be issued in the near future.

IV FUTURE WORK

A. Joining of the major subassemblies of the Prototype Corrosion Loop will be completed, and instrumentation of the system at the test site will be performed.

B. The potassium for the loop will be purified and both the sodium, and the potassium transfer system will be flushed out prior to filling and flushing the loop circuits.

C. Following closing of the test chamber, the system will be baked out at 500°F under vacuum for several hundred hours while low temperature checkout and component calibration tests are being performed. Present schedules indicate that the test conditions should be attained near the end of the next reporting period.

REFERENCES

(1) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 6 for Period Ending January 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54344, p. 16.

(2) Personal Communication from Dr. E. L. Steele.

(3) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 5 for Period Ending October 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54269, p. 17.

(4) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 4 for Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167, p. 9.

(5) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 4 for Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167, p. 30.

(6) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 6 for Period Ending January 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54344, p. 19.

(7) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 3 for Period Ending April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54081, p. 11.

(8) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 5 for Period Ending October 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54269, p. 7.

(9) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 6 for Period Ending January 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54344, p. 29.

(10) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 6 for Period Ending January 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54344, p. 29.

(11) Semi-annual Progress Report, Determination of the Emissivity of Materials, Report Period: May 14-Nov. 15, 1964, R. C. Emanuelson, Pratt & Whitney Aircraft, NASA Contract NAS 3-4174, NASA-CR-54268.

(12) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 6 for Period Ending January 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54344, p. 36.

REFERENCES (Cont'd)

(13) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 4 for Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167, p. 43.

(14) Design of Piping Systems, John Wiley & Sons, New York, 1956, p. 221.

(15) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 5 for Period Ending October 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54269, p. 28.

(16) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 6 for Period Ending January 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54344, p. 44.

(17) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 5 for Period Ending October 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54269, p. 43.

(18) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 5 for Period Ending October 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54269, p. 17.

(19) Ewing, C. T., Stone, J. P., Spann, J. R., Steinkuller, E. W., Williams, D. D., and Miller, R. R., "High Temperature Properties of Sodium and Potassium," NRL Report 6128, August, 1964.

REPORT DISTRIBUTION LIST - Contract NAS3-2547
Quarterly and Final

NASA
Washington, D.C., 20546
Attn: James J. Lynch (RN)

NASA
Washington, D.C., 20546
Attn: George C. Deutsch (RR)

NASA
Scientific & Technical Information Facility
Box 5700
Bethesda 14, Maryland
Attn: NASA Representative (2 copies + 2 repro.)

NASA
Ames Research Center
Moffet Field, California
Attn: Librarian

NASA
Goddard Space Flight Center
Greenbelt, Maryland
Attn: Librarian

NASA
Langley Research Center
Hampton, Virginia
Attn: Librarian

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Librarian MS 3-7

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Dr. Bernard Lubarsky MS 86-1

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Roger Mather (500-309)

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: G. M. Ault MS 105-1

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: John E. Dilley MS 86-1

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: T. A. Moss MS 86-5

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: R. L. Davies MS 86-5 (5)

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Dr. Louis Rosenblum MS 106-1

NASA-Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Technology Utilization Officer MS 3-16

NASA
Manned Spacecraft Center
Houston 1, Texas
Attn: Librarian

NASA
George C. Marshall Space Flight Center
Huntsville, Alabama 35812
Attn: Librarian

NASA
Jet Propulsion Laboratory
4800 Oak Grove
Pasadena, California 91103
Attn: Librarian

NASA
Western Operations Office
150 Pico Boulevard
Santa Monica, California 90406
Attn: John Keeler

National Bureau of Standards
Washington 25, D.C.
Attn: Librarian

Flight Vehicle Power Branch
Air Force Aeropropulsion Laboratory
Wright Patterson Air Force Base, Ohio
Attn: Charles Armbruster ASRPP-10

Flight Vehicle Power Branch
Air Force Aeropropulsion Laboratory
Wright Patterson Air Force Base, Ohio
Attn: T. Cooper

Flight Vehicle Power Branch
Air Force Aeropropulsion Laboratory
Wright Patterson Air Force Base, Ohio
Attn: Librarian

Flight Vehicle Power Branch
Air Force Aeropropulsion Laboratory
Wright Patterson Air Force Base, Ohio
Attn: George M. Glenn

Flight Vehicle Power Branch
Air Force Aeropropulsion Laboratory
Wright Patterson Air Force Base, Ohio
Attn: Mr. George Sherman API

Flight Vehicle Power Branch
Air Force Aeropropulsion Laboratory
Wright Patterson Air Force Base, Ohio
Attn: Mr. George E. Thompson APIP-1

Army Ordnance Frankford Arsenal
Bridesburg Station
Philadelphia 37, Pennsylvania
Attn: Librarian

Bureau of Mines
Albany, Oregon
Attn: Librarian

Bureau of Weapons
Research and Engineering
Material Division
Washington 25, D.C.
Attn: Librarian

U.S. Atomic Energy Commission
Technical Reports Library
Washington 25, D.C.
Attn: J. M. O'Leary

U.S. Atomic Energy Commission
Germantown, Maryland
Attn: Mr. H. Finger

U.S. Atomic Energy Commission
Germantown, Maryland
Attn: H. Rochen

U.S. Atomic Energy Commission
Germantown, Maryland
Attn: Major Gordon Dicker
SNAP 5-/SPUR Project Office

U.S. Atomic Energy Commission
Technical Information Service Extension
P.O. Box 62
Oak Ridge, Tennessee (3)

U.S. Atomic Energy Commission
Washington 25, D.C.
Attn: M. J. Whitman

Argonne National Laboratory
9700 South Cross Avenue
Argonne, Illinois
Attn: Librarian

Bureau of Ships
Department of Navy
Washington 25, D.C.
Attn: Librarian

Brookhaven National Laboratory
Upton, Long Island, New York
Attn: Librarian

Report Distribution List NAS3-2547 - Quarterly and Final - (Continued)

Oak Ridge National Laboratory
Oak Ridge, Tennessee
Attn: W. O. Harms

Oak Ridge National Laboratory
Oak Ridge, Tennessee
Attn: Dr. A. J. Miller

Oak Ridge National Laboratory
Oak Ridge, Tennessee
Attn: Librarian

Oak Ridge National Laboratory
Oak Ridge, Tennessee
Attn: J. H. DeVan

Office of Naval Research
Power Division
Washington 25, D.C.
Attn: Librarian

U.S. Naval Research Laboratory
Washington 25, D.C.
Attn: Librarian

Advanced Technology Laboratories
Division of American Standard
360 Whisman Road
Mountain View, California
Attn: Librarian

Aerojet-General Corporation
P.O. Box 296
Azusa, California 91703
Attn: R. S. Carey

Aerojet-General Corporation
P.O. Box 296
Azusa, California 91703
Attn: Librarian

Oak Ridge National Laboratory
Oak Ridge, Tennessee
Attn: G. Goldberg

Aerojet General Nucleonics
P.O. Box 77
San Ramon, California
Attn: Librarian

AiResearch Manufacturing Company
Sky Harbor Airport
402 South 35th Street
Phoenix, Arizona
Attn: Librarian

AiResearch Manufacturing Company
Sky Harbor Airport
402 South 35th Street
Phoenix, Arizona
Attn: E. A. Kovacevich

AiResearch Manufacturing Company
9851-9951 Sepulveda Boulevard
Los Angeles 45, California
Attn: Librarian

Allis Chalmers
Atomic Energy Division
Milwaukee, Wisconsin
Attn: Librarian

Allison-General Motors
Energy Conversion Division
Indianapolis, Indiana
Attn: Librarian

AMF Atomics
140 Greenwich Avenue
Greenwich, Connecticut
Attn: Librarian

Armour Research Foundation
10 W. 35th Street
Chicago 16, Illinois
Attn: Librarian

Atomics International
8900 DeSoto Avenue
Canoga Park, California
Attn: Librarian

AVCO
Research and Advanced Development
Department
201 Lowell Street
Wilmington, Massachusetts
Attn: Librarian

Babcock and Wilcox Company
Research Center
Alliance, Ohio
Attn: Librarian

Battelle Memorial Institute
505 King Avenue
Columbus, Ohio
Attn: Librarian

The Boeing Company
Seattle, Washington
Attn: Librarian

Brush Beryllium Company
Cleveland, Ohio
Attn: Librarian

Carborundum Company
Niagara Falls, New York
Attn: Librarian

Chance Vought Aircraft, Incorporated
P.O. Box 5907
Dallas 22, Texas
Attn: Librarian

Climax Molybdenum Company of Michigan
1600 Huron Pky.
Ann Arbor, Michigan 48105
Attn: Librarian

Convair Astronautics
5001 Kerrny Villa Road
San Diego 11, California
Attn: Librarian

Crucible Steel Company of America
Pittsburgh, Pennsylvania
Attn: Librarian

Curtiss-Wright Corporation
Research Division
Quenanna, Pennsylvania
Attn: Librarian

Douglas Aircraft Company
Santa Monica, California
Attn: Librarian

E. I. duPont de Nemours and Company, Inc.
Wilmington 98, Delaware
Attn: Librarian

E. I. duPont de Nemours and Company, Inc.
Wilmington 98, Delaware
Attn: H. Hix

Electro-Optical Systems, Incorporated
Advanced Power Systems Division
Pasadena, California
Attn: Librarian

Fansteel Metallurgical Corporation
North Chicago, Illinois
Attn: Librarian

Firth Sterling, Incorporated
McKeesport, Pennsylvania
Attn: Librarian

Ford Motor Company
Aeronutronics
Newport Beach, California
Attn: Librarian

General Atomic
John Jay Hopkins Laboratory
P.O. Box 608
San Diego 12, California
Attn: Librarian

General Atomic
John Jay Hopkins Laboratory
P.O. Box 608
San Diego 12, California
Attn: Dr. Ling Yang

General Electric Company
Atomic Power Equipment Division
P.O. Box 1131
San Jose, California

General Electric Company
Missile and Space Vehicle Department
3198 Chestnut Street
Philadelphia 4, Pennsylvania
Attn: Librarian

General Electric Company
P.O. Box 100
Richland, Washington 99352
Attn: Technical Information Operation

General Electric Company
P.O. Box 100
Richland, Washington 99352
Attn: Dr. T. T. Claudson

General Electric Company
Vallecitos Atomic Laboratory
Pleasanton, California
Attn: Librarian

General Dynamics/Fort Worth
P.O. Box 748
Fort Worth, Texas
Attn: Librarian

General Motors Corporation
Allison Division
Indianapolis 6, Indiana
Attn: Librarian

Grumman Aircraft
Bethpage, New York
Attn: Librarian

Hamilton Standard
Division of United Aircraft Corporation
Windsor Locks, Connecticut
Attn: Librarian

Hughes Aircraft Company
Engineering Division
Culver City, California
Attn: Librarian

Lockheed Georgia Company
Division, Lockheed Aircraft Company
Marietta, Georgia
Attn: Librarian

Lockheed Missiles and Space Division
Lockheed Aircraft Corporation
Sunnyvale, California
Attn: Librarian

Los Alamos Scientific Laboratory
University of California
Los Alamos, New Mexico
Attn: Librarian

Marquardt Aircraft Company
P.O. Box 2013
Van Nuys, California
Attn: Librarian

The Martin Company
Baltimore 3, Maryland
Attn: Librarian

The Martin Company
Nuclear Division
P.O. Box 5042
Baltimore 20, Maryland
Attn: Librarian

Martin Marietta Corporation
Metals Technology Laboratory
Wheeling, Illinois

Massachusetts Institute of Technology
Cambridge 39, Massachusetts
Attn: Librarian

Materials Research Corporation
Orangeburg, New York
Attn: Librarian

McDonnell Aircraft
St. Louis, Missouri
Attn: Librarian

MSA Research Corporation
Callery, Pennsylvania
Attn: Librarian

National Research Corporation
405 Industrial Place
Newton, Massachusetts
Attn: Librarian

North American Aviation
Los Angeles Division
Los Angeles 9, California
Attn: Librarian

Pratt and Whitney Aircraft
400 Main Street
East Hartford 8, Connecticut
Attn: Librarian

Republic Aviation Corporation
Farmingdale, Long Island, New York
Attn: Librarian

Rocketdyne
Canoga Park, California
Attn: Librarian

Solar
2200 Pacific Highway
San Diego 12, California
Attn: Librarian

Southwest Research Institute
8500 Culebra Road
San Antonio 6, Texas
Attn: Librarian

Superior Tube Company
Morristown, Pennsylvania
Attn: Mr. A. Bounds

Sylvania Electrics Products, Inc.
Chem. and Metallurgical
Towanda, Pennsylvania
Attn: Librarian

Thompson Ramo Wooldridge, Inc.
Caldwell Research Center
23555 Euclid Avenue
Cleveland, Ohio 44117
Attn: Librarian

Thompson Ramo Wooldridge, Inc.
Caldwell Research Center
23555 Euclid Avenue
Cleveland, Ohio 44117
Attn: G. J. Guarnieri

Thompson Ramo Wooldridge, Inc.
New Devices Laboratories
7209 Platt Avenue
Cleveland, Ohio 44104
Attn: Librarian

Union Carbide Corporation
Parma Research Center
Technical Information Service
P.O. Box 6116
Cleveland, Ohio 44101

Union Carbide Stellite Corporation
Kokomo, Indiana
Attn: Librarian

Union Carbide Nuclear Company
P.O. Box X
Oak Ridge, Tennessee
Attn: X-10 Laboratory Records
Department (2)

United Nuclear Corporation
Five New Street
White Plains, New York
Attn: Librarian

Universal Cyclops Steel Corporation
Refractomet Division
Bridgeville, Pennsylvania
Attn: C. P. Mueller

University of Michigan
Department of Chemical and
Metallurgical Engineering
Ann Arbor, Michigan
Attn: Librarian

Vought Astronautics
P.O. Box 5907
Dallas 22, Texas
Attn: Librarian

Wah Chang Corporation
Albany, Oregon
Attn: Librarian

Westinghouse Electric Corporation
Astronuclear Laboratory
P.O. Box 10864
Pittsburgh 36, Pennsylvania
Attn: Librarian

Report Distribution List NAS3-2547 - Quarterly and Final - (Continued)

Westinghouse Electric Corporation
Astronuclear Laboratory
P.O. Box 10864
Pittsburgh 36, Pennsylvania
Attn: R. T. Begley

Westinghouse Electric Corporation
Materials Manufacturing Division
RD #2 Box 25
Blairsville, Pennsylvania
Attn: F. L. Orell

Westinghouse Electric Corporation
Materials Manufacturing Division
RD #2 Box 25
Blairsville, Pennsylvania
Attn: Librarian

Westinghouse Electric Corporation
Aerospace Electrical Division
Wapak Road
Lima, Ohio
Attn: Paul Kueser

American Machine and Foundry Company
Alexandria Division
1025 North Royal Street
Alexandria, Virginia
Attn: Librarian

Douglas Aircraft Company, Inc.
Missile and Space Systems Division
300 Ocean Park Boulevard
Santa Monica, California
Attn: Librarian

Westinghouse Electric Corporation
Aerospace Electrical Division
Lima, Ohio
Attn: Librarian

Westinghouse Electric Corporation
Research and Development Laboratory
Pittsburgh 35, Pennsylvania

Ertel-McCullough, Inc.
301 Industrial Way
San Carlos, California
Attn: Dr. Leonard Reed

Mr. Rudolph Rust - MS 138-214
Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, California 91102

Mr. W. H. Podolny
United Aircraft Corporation
Pratt & Whitney Division
400 W. Main Street
Hartford 8, Connecticut

Varian Associates
Vacuum Products Division
611 Hansen Way
Palo Alto, California
Attn: J. Shields

Ultek Corporation
920 Commercial Street
Palo Alto, California
Attn: Librarian